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ABSTRACT

This Records and Archives Management Programme (RAMP) study covers the conservation of archival documents and the application of freeze-drying to the salvage of documents damaged by flood. Following an introductory discussion of the hazards of water, the study presents a broad summary of data on freeze-drying, including the behavior of water-soaked paper; the advantages of stabilization by freezing; descriptions of vacuum freeze-drying and alternate methods of drying; and a comparison of two currently used methods (1) vacuum freeze-drying by sublimation (a dry process) and (2) vacuum-drying by evaporation (a wet process). The history of freeze-drying is described; six case histories illustrating the use of vacuum chambers for the recovery of water-damaged archival and library materials are presented; and examples of the use of vacuum and freeze-drying in eight countries are summarized. Also considered are commercial sources for freezing and drying; the cost of vacuum-drying and freeze-drying; low-cost freezing and drying in an emergency; what freeze-drying will not do; and cooperative approaches to the use of freeze-drying chambers. Finally, 24 imaginary open forum questions and answers related to freeze-drying are presented, and disaster preparedness planning is discussed. The text is supplemented with two figures and two tables, and a 75-item bibliography is provided. (KM)

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Vacuum freeze-drying, a method used to salvage water-damaged archival and library materials: a RAMP study

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VACUUM FREEZE-DRYING, A METHOD USED TO SALVAGE WATER-DAMAGED ARCHIVAL AND LIBRARY MATERIALS: A RAMP STUDY WITH GUIDELINES

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PREFACE

In order to assist in meeting the needs of Member States, particularly developing countries, in the specialized areas of Archives Administration and Records Management, the Division of the General Information Programme has developed a long-term Records and Archives Management Programme - RAMP.

The basic elements of RAMP reflect and contribute to the overall themes of the General Information Programme. RAMP thus includes projects, studies and other activities intented to :

- develop standards, rules, methods and other normative tools for the processing and transfer of specialized information and the creation of compatible information systems ;

- enable developing countries to set up their own data bases and to have access to those now in existence throughout the world, so as to increase the exchange and flow of information through the application of modern technologies ;

- promote the development of specialized regional information networks ;

- contribute to the harmonious development of compatible international information services and systems ;

- set up national information systems and improve the various components of these systems ;

- formulate development policies and plans in this field ;

- train information specialists and users and develop the national and regional potential for education and training in the information sciences, library science and archives administration.

This RAMP study covers the conservation of archival documents and the application of freeze-drying to the salvage of documents damaged by flood. As indicated by the author, the study summarizes a broad spectrum of data on freeze-drying and cites two methods in current use for drying water-damaged archival and library material, gives ideas on disaster-preparedness planning and identifies recommended literature which is given in the list of references accompanying the study.

Comments and suggestions regarding the study are welcomed and should be addressed to the Division of tthe General Information Programme, UNESCO, 7, Place de Fontenoy, 75700 Paris, France. Other studies prepared as part of the RAMP programme may also be obtained at the same address.

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FOREWORD

Freeze-drying is a familiar term thanks to the propaganda given certain consumer foods and beverages available on the market. These vary from dehydrated strawberries to brownish crystals in a jar that with hot water are converted to a cup of coffee. Some people may know that such products are made in a vacuum chamber, the same type as those used as simulators for training astronauts, or testing satellites and moon vehicles before launching them off into space. But, mention a vacuum chamber or simulator as an effective tool for drying a ton of documents soaked by a broken water main, or stacks of books soaked by the fire hoses used in fighting a blaze, and the number of those up-to-date diminishes.

Among archivists and librarians are many who remember "reading something about it" in professional periodicals, but retain a fleeting idea of what the process is, how it works, how it applies to them. Hence, the purpose of this study: to fill the information gap. To this end, the study summarizes a broad spectrum of data that includes, among other relevant facts, the behavior of paper when wet, its vulnerabilities in this condition, stabilization by freezing, vacuum chambers and how they dry, and selected case histories where such chambers were used successfuly to salvage water-damaged documents and books.

The study cites two methods in current use for drying water-damaged archival and library materials: vacuum freeze-drying by sublimation and vacuum-drying by evaporation. The emphasis is on freeze-drying. Here, the wet materials must first be frozen. This produces a condition of total stabilization which, in turn, provides un unlimited span of time to think, plan, and decide what course of action to take. These advantages, however, do not indicate that vacuum-drying is automatically downgraded. As the study reveals, it is a very effective method of drying. But, since the materials normally are not prefrozen, certain exigencies could arise. For example, depending on conditions, wet materials might have to be placed in a chamber as quickly as possible to prevent attack by mold.

Many people - conservators, conservator-scientists, and manufacturers - kindly furnished information on freeze-drying in response to letters written to them by the author. They warrant profuse thanks and gratitude for the time they took from their busy schedules to provide a reply.

1. INTRODUCTION

1.1 Water: The Ubiquitous Hazard

The primary enemies of documents housed in archives and libraries have always been fire and water, each an agent of a specific type of destruction. Although fires, with their spectacular display and awesome finality, are thought to be the predominate cause of destruction, in reviewing the literature on disasters in archives and libraries, the majority appears to be the result of water. In a fire-gutted building the destroyed holdings may have to be written off. On the other hand, although water can, under certain conditions, be just as destructive the materials can be saved if appropriate action is taken.

Of the many recorded reports on disasters where water used to extinguish a fire did as much or more damage than the fire, there are two striking examples. The first was witnessed by Schmelzer (1), librarian of the Library of the Jewish Theological Seminary in New York City. In November 1966 a fire broke out in one of the top floors of the ten story building. As a result some 70,000 books were practically burned to ashes. However, because of the open multitiered stack structure of the library, the water used to extinguish the fire poured down seven floors and damaged the remaining 150,000 volumes.

The second case, reported by Stender and Walker (2), involved one of the largest archives known: The National Personnel Records Office in South St Louis, Missouri, which contains millions of individual records of former federal government employees, both military and civilian, in a six story building of solid concrete. In July 1963 a fire broke out on the sixth floor. Through a continuous flood of water, the firemen were successful in confining the fire to that level. But after the fire was extinguished, all six levels of the center had several inches of water on the floor. Water flowed freely through the building, especially through utility areaways and points where internal pipes ran through the floor. Broken water lines on the sixth floor added to the flood conditions. Some 10,000 cubic feet (280 cubic meters) of water-damaged records were removed for treatment from the lower flooded floors along with the several thousands of cubic feet or records from the sixth floor that suffered both fire and water-damage.

Although the water used to extinguish a fire can wreak destruction greater than the fire, this source of misplaced water is, fortunately, not too occasional thanks to the construction of fire-proof buildings and professional firefighters (3). On the other hand, there are many other sources of water that constitute a potential hazard to collections in archives and libraries. One source can be attributed to nature; tornados, hurricanes, floods, rainstorms, snow. The other to man: leaks in water supply or drainage pipes; breaks and/or leaks in steam pipes in the building ; the breakdown of water heaters or air conditioning systems; clogged sinks, basins

toilets; leakage of water deposits or tanks; leakage of roofs and windows; seepage in basements; clogged roof gutters and downpipes; broken water mains.

A classic example of the damage a broken water main can cause is found in the well-documented account of the Stanford University flood (4). On November 8, 1978, an eight inch (25.4 centimeters) water main ruptured outside of one of the Stanford libraries - the Meyer Research Library - where new construction was underway. The rupture site was some 20 feet from the nearest wall. The flow of water was turned off about 20 minutes after the rupture took place. In the meantime, water entered the basement of the library where there were two levels of a metal-tiered construction and damaged some 50,000 research volumes.

2. WATER-SOAKED PAPER

2.1 Associated Problems

2.1.1 Absorption and Swelling

Paper has a normal hygroscopic capacity for absorbing water. However, paper made before the middle of the nineteenth century has an even greater capacity for the absorption of water thanks to the greater amount of water-soluble sizing used in that early period. For example, books of this period will absorb up to an average of 80 percent of their original weight. Furthermore, paper of this period used for books or manuscripts is highly vulnerable to microbiological infection. However, such paper will survive total immersion in water for longer periods than paper made after the mid-nineteenth century. Books made after this time were treated with water-resisting sizes; they absorb an average of 60 percent of their weight. So, in estimating the original weight of a collection, if each book weighs about four pounds (1.81 kilograms) when dry and there are, for example, about 20,000 of each period, one must plan for the removal of 64,000 pounds (29,000 kilograms) of water from the earlier period and 48,000 pounds (21,773 kilograms) from the later (5).

As to swelling of books, the major part of damage takes place within the first eight hours after soaking.And since the text block and book covers swell more than the covering material, the tensions produced causes the spine to become concave and the fore-edge convex. The straining forces the case of the book to become partially or completely detached (5).

2.1.2 Microbiological Infection

Gallo (6) describes a number of schizomycetes (bacteria), actually low in number, and about 100 species of fungi which, under favorable conditions, attack and infest the organic matter in paper. Although the spores of the fungi and the schizomyceti are present in the raw materials used for the manufacturing of paper waiting for conditions favorable for development, the infection of documents is more attributable to the spores ever present in the air or dust. Spores need air to develop; a book or a bundle of manuscripts totally immersed in water are immune from attack. And since the spores of the fungi cause more frequent and greater damage than bacteria, this study will focus on the former.

The microbial spores of the fungal plant, of which the most common is called mold, need the following elements in order to reproduce: humidity, a relatively warm temperature, and a nutrient. The first, humidity, is present in abundance when water floods an archives or library. And a temperature higher than normal is present if the season is warm (the problem is less acute if it is cold), if ventilation is poor, if the air conditioning system breaks down, or if there is heat present generated by an extinguished fire. The third element, the nutrient, is essential because the fungal plant, which has no chlorophyll to convert carbon dioxide to carbohydrates for tissue growth, must get its carbohydrates directly from organic matter. Unfortunately, there is plenty of nutrition available in the cellulose of paper, the protein in parchment and leather, along with the nutrients in animal and starch adhesives used to size, glue and paste.

Kowalik (7) adds that microorganisms not only have cellulose at their disposition but also other substances such as lignin, hemicelluloses, pectins, waxes, tannin, and mineral constituents. Furthermore, paper may contain resinous fillers, dyestuffs, added during the productive cycles, and various impurities which may also form a part of the microbial diet.

In any case, if the temperature is in the 18° to 36°C (65° to 96.8°F) range and the relative humidity above 65 percent, chances are that mold infection will appear on wetted books and documents in about 72 hours after flooding takes place. This, however, is not a hard and fast rule. Waters (8) reports a case where mold developed rampantly on the water-soaked spines of rare books to a thickness of 1/4 to 1/2 inch (6 to 12.7 millimeters) some 52 hours from the onset of a disaster. As a matter of fact, during long-term storage fungi will grow, although slowly, at a relative humidity as low as 60 percent. And recently the National Library of Wales (9) experienced mold infection (no flood involved) on a number of items where the relative humidity was not only well below 70 precent, but in some cases as low as 50 percent.

The first visible evidence of mold infection is a white powdery mass that will appear on the surface of a document or a book. Sometimes you need a raking light to detect it. Even a slight trace is a warning that temperature and humidity are above the limits for safety. At this point, the fungal plant has grown hyphae (root-like organs) into the stratum of the nutrient in order to get food for its development. As the plant metabolizes the substances required for its growth, it secretes citric, oxalic, lactic, and other organic acids which damage the material on which the mold is feeding. At the same time, the plant secretes pigments of green, blue, brown, black, red, and yellow color which are deposited on the host. These stains are practically impossible to remove ; they can obliterate the text of a manuscript or book.

When mold attacks paper other complex things happen: the strength supplied by the sizing in paper diminishes; when cellulose suffers the attack, the structure of the paper is damaged to the point that it will become soft or so fragile that it will actually break.

Gallo (6) points out that in some cases fungi can exercise a mechanical action on paper: their hyphae may filtrate between fibers of the paper without actually entering them, or the fruit bodies of the fungi, which are covered with bristles, infiltrate between one leaf and another; in both cases the pages of a book or documents in a bundle are welded together.

2.1.3 Adhesion of Leaves

When a bundle of documents or a book has been soaked and permitted to dry under favorable conditions, it will begin to lose its water content from the outside surface. Capillary action permits the interior water to move outward and carry with it all varieties of water-soluble materials such as dyes, pigments, adhesives, and acids. In a book, the concentration of these materials, the acids and adhesives in particular, will cause the edges of the text block to become embrittled and edges stick together. If these text blocks are left in this condition after drying, serious degradation of the cellulose in the paper will be speeded up (8).

Books on coated paper present special problems. Coatings are usually applied to paper in order to obtain uniformity of surface, to enhance opacity, smoothness, and gloss. The basic components of coatings contain pigments such as China clay or a solubilized protein (10). Waters (8) notes that in the presence of water, starch-based coatings and some casein mixtures may revert from dry adhesive to gel and then back to the solid on drying. When these adhesive mixtures are in a fluid state, any pressure will cause the coatings to weld together and create a permanent bond during the drying cycle.

2.1.4 Migration of Inks and Dyes

In case of wetted hand-written documents or bound volumes where inks have been used, the archivist and librarian face the problem of feathering or migration of those inks. In the two principal categories of inks, those made from carbon pose no problem since this organic material is not soluble: its binding medium (glue or gum), even when long decayed, leaves the carbon particles embedded in the paper fibers. However, there are inks that may look like carbon but are, in fact, quite soluble. In the second principal category are the so-called iron inks which are compounded from gallotannic acid in the presence of iron in a binding medium. Where carbon remains on the surface, iron inks soak into the paper so that the insoluble iron compounds formed when the ink matures are held as an integral part of the paper surface. In those cases where mature iron inks become a rusty brown or yellowish color it is not safe to assume that all inks of these tones are of iron and therefore insoluble. These same colors can appear with inks made of sepia or beechwood root which are impermanent (11).

Through all times inks have been made according to a multitude of recipes, and as a result vary in substance, appearance, and permanence. Some more modern inks are made "permanent" for fountain pens by using iron sulphate and tannic acid. However, the dyes used for toning will feather in water. Some inks used for writing have little more than a dilute aqueous solution of one or more synthetic dyestuffs. Other "permanent" inks like those made of iron compounds will run or feather in water until they are matured or, said in another manner, properly oxidized. As for colored inks, most of them are soluble with or without aging.

While most holdings in archives and libraries are of non-colored paper, the majority of the more modern material, even if it appears to be white, contains coloring matter added to improve appearance. For toning or coloring there are two major classes of colorants used: colored pigments and water soluble dyes. Of these the latter are the most used. The word "soluble" means that a dye is soluble in water and colors the fibers from a solution in water. Such colors are impermanent and will run when wetted.

2.1.5 Time

The problems associated with water damage to archival and library materials - absorption and swelling, mold infection, blocking or adherence of leaves, migration of inks and dyes - get worse with the passage of time; salvage becomes more difficult, costs may go up. Future repairs and restoration may become more complicated, more expensive and time consuming. In short, time is a problem if it is not available. However, there is a way to by an unlimited amount: you do it with a process called stabilization by freezing.

3. STABILIZATION BY FREEZING

The most effective method found to date, and the most generally accepted by conservators for the stabilization of water-damaged archival and library materials is freezing at low temperatures. A level of about -30°C (-20°F) is recommended; the frozen materials remain in cold storage. Quick freezing, such as the so-called blast freezing, is a method used to produce the formation of the smallest ice crystals possible. Slow freezing produces large, needlelike crystals.

3.1 Advantages

3.1.1 Halts Mold Attack

Without the conditions required for the reproduction and development of the spores, mold infection ceases upon freezing. True, the mold spores are not destroyed by freezing; they remain in a dormant state until a more favorable environment is available. But the fact remains that the infection is effectively checked and its pernicious damage cannot take place.

3.1.2 Stabilizes Soluble Inks and Dyes

Freezing has the additional advantage of stabilizing inks, dyes, dyestuffs, colorants, etc, used for manuscripts, maps, sketches, drawings, and such, that are soluble in water. Later, when freeze-drying takes place, migration or feathering of inks or dyes can be restrained since the liquid stage is by-passed.

3.1.3 Prevents Adhesion of Leaves

The problem of blocking or adhesion of leaves to each other is primarily confined to books and periodicals printed on stock that uses a coating pigment with a binder of casein and starch, two highly water-soluble substances. If wetted material of coated stock is permitted to dry it will turn a book to a clay-like brick. Restoration is impossible. At the moment the only known salvage method considered practical, especially where large quantities of water-damaged material are involved, is freezing whilewet then freeze-drying.

3.1.4 Permits Orderly, Unhurried Planning

The freeze stabilization of water-damaged archival and library materials lifts a tremendous load from the shoulders of those who have suffered a disaster. Stabilization permits time to confer with experts on the selection of drying methods. There is time to assess damage, to see what can be discarded, replaced, microfilmed; time to determine what repairs or restoration might be required; time to rehabilitate a damaged storage area, or to find an alternate location.

4. VACUUM FREEZE-DRYING

4.1 A Primer on Freeze-Drying

4.1.1 Units of Measurement

First, it may be helpful to review the terms used commonly to express the amount of pressure to which the vacuum chamber used for drying is subjected. As the reader will note further on, freeze-drying is one of the processes carried out in a vacuum; the conditions are below normal atmospheric pressure. Normal (sea level) atmospheric pressure is 1.033 kilograms per square centimeter, or 14.7 pounds per square inch, 760 torr, or 1,000 millibars. In scientific parlance, 1 atmosphere is equivalent to 760 torr (1 torr is equivalent to 1 millimeter of mercury whose symbol is Hg). For many years the unit of measurement for pressure was in mm Hg. Then, in recognition of Torrecelli's work - the invention in 1843 of the mercury barometer - the unit was renamed the torr. However, the two terms, mm Hg and torr, are current and will be used interchangeably in this study.

As a matter of information, the unit of measurement in the Systeme Internationale D'Unites (SI System) is the Pascal which is related to the bar. Another unit used but considered obsolescent is the micron.

Torr	mm Hg	Micron	Pascal	Millibar
1	1	1,000	133.3	1.333
10^{-1}	0.1	100	13.33	0.133
10^{-2}	0.01	10	1.33	0.0133
10^{-3}	0.001	1	0.133	0.00133

Table 1. Typical Units Used to Measure Vacuum Pressure.

4.1.2 Sublimation/Evaporation

For the purpose of this study, freeze-drying is defined as a method of drying wetted archival and library materials by freezing, then under vacuum conditions by converting the solid to the vapor phase; the liquid phase is by-passed.

When a liquid is converted to the vapor phase by heating, the action is called evaporation. As a solid (frozen), its conversion to the vapor phase by the application of heat without going through the liquid phase is called sublimation. An example of sublimation: preservation in high mountainous regions where water in meat freezes then evaporates (sublimes) with no liquid phase to produce unwanted side effects.

The process of both sublimation and evaporation depends ultimately on the relationship of temperature and pressure to the kinetic energy (energy of constant motion) present in water as a liquid or solid. For example, in a kettle of water, as the temperature is increased the kinetic energy increases and permits molecules from the liquid to escape as vapor. Maximum vaporization occurs at the boiling point, 100°C (212°F) at 1 atmosphere. Conversely, the vapor pressure of water, that is, the pressure of the vapor in equilibrium with the liquid, is 760mm Hg at 100°C. If the atmospheric pressure is reduced to say, 525.8mm Hg, as would be the case if the same kettle is boiled on the top of a mountain, the boiling point will be reduced by several degrees to 90°C (194°F). The reason: at this temperature the vapor pressure of water is 525.8mm Hg.

Water as a solid behaves in much the same way as water as a liquid except that the temperature at which the energy of motion of the solid (ice) permits the release of water vapor molecules begins at the opposite end of the thermometer scale: the freezing point and below. The vapor pressure scales become numerically very low. By using selected temperatures below the freezing point together with suitable pressures, the sublimation of the solid will take place. This will be explained later in the text.

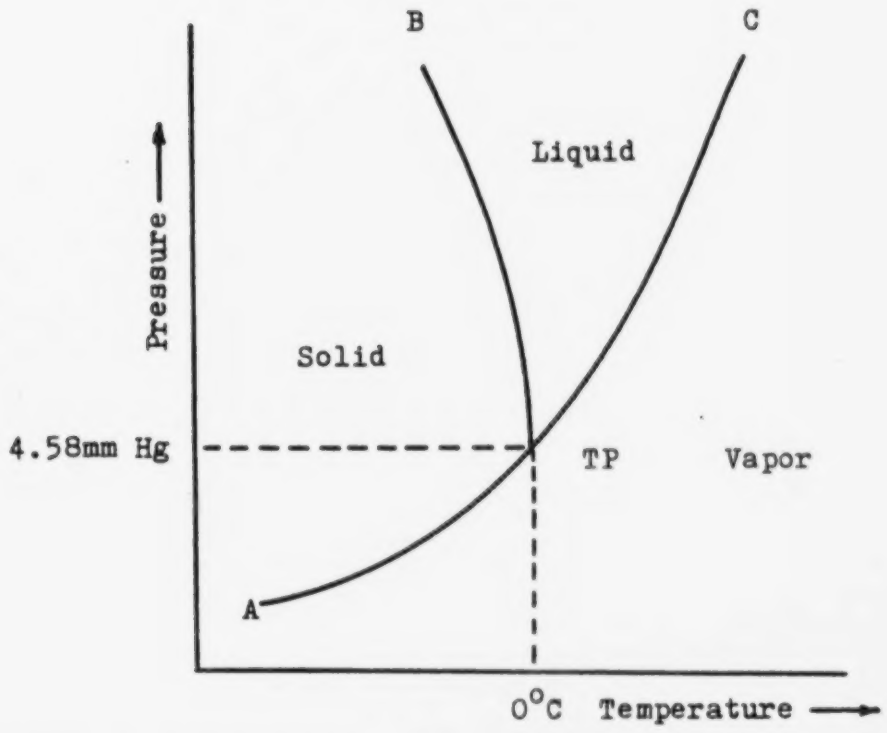

Figure 1. Temperature-Pressure Equilibrium Curves of Pure Water.

4.1.3 Temperature-Pressure Values of Water

A graphic method of explaining the temperature-pressure influence in water as a solid, a liquid, and a vapor is in the temperature-pressure equilibrium curves of pure water shown in Figure 1. At the triple point TP, at a pressure of 4.58mm Hg and a temperature of 0°C (32°F), the three states of water - solid, liquid, vapor - can exist in a state of equilibrium. The solid exists in the area between A-TP and B-TP; the liquid between B-TP and C-TP; the vapor below the area A-TP-C.

A point on any curve represents a condition of equilibrium between any two states at a definite temperature and pressure. If the temperature is raised and pressure is increased the solid enters the liquid zone and melts. And if the temperature is reduced and pressure held constant or reduced the solid can become vapor. In other words, sublimation becomes possible because there is no "liquid" zone between the "solid" and "vapor" zones. If the system varies in either temperature or pressure from the triple point value at least one of the three states disappears.

°C	Torr	°C	Torr	°C	Torr
100	760.0	0	4.58	-24	.53
60	149.4	-2	3.88	-26	.43
50	92.51	-4	3.28	-30	.29
40	55.32	-6	2.76	-32	.23
30	31.82	-8	2.33	-34	.19
20	17.54	-10	1.95	-36	.15
10	9.21	-12	1.63	-38	.12
5	6.54	-14	1.36	-40	.097
4	6.10	-16	1.13	-42	.077
3	5.69	-18	.94	-44	.061
2	5.29	-20	.78	-50	.0296
1	4.93	-22	.64	-60	.0081

Table 2. Vapor Pressure/Temperature Relationship for Water/Ice.

4.1.4 Vapor Pressure

Ice within a chamber evacuated of air and maintained at a given temperature will give off molecules in the form of water vapor. This escape (sublimation) is continuous. The molecules will bump into each other and the chamber wall; some will return to the surface of the ice. Eventually, the rate at which the molecules return to the ice will equal the rate at which they leave the ice. At this point, the water vapor and ice are in a state of equilibrium; the pressure at which this occurs is called the equilibrium vapor pressure. If the temperature in the chamber is, say, -20°C (-4°F) the pressure will reach 0.78mm Hg. This is the equilibrium vapor pressure and, consequently, the vapor pressure as seen in Table 2. The pressure exerted by vapor depends only on temperature as long as there is some liquid present.

4.2 Conditions Required for Freeze-Drying

First, the object must be in a frozen state from which, as we have seen, water vapor molecules escape. Next, in order to induce continued sublimation a method must be found which will remove the water vapor molecules in the vicinity of the ice. The most effective method is to draw the molecules away by creating a colder surface (condenser) with a lower vapor pressure elsewhere, but near the vicinity of the ice. Under this condition water vapor molecules will diffuse to the colder surface where they will recondense and remain trapped as ice crystals.

To illustrate this frozen object-condenser relationship: if the chamber where the frozen object rests has a temperature of, for example, -14°C (6.80°F) its vapor pressure is 1.36mm Hg according to Table 2. If the temperature of the nearby condenser is -40°C (-40°F), the vapor pressure on the cold surface is 0.097mm Hg. This is equivalent to a vapor pressure ratio of 14 to 1, a difference that will go far in drawing water vapor away from the frozen object.

The next step is to find a method of providing an easier path for the water vapor molecules to travel on the way to the condenser trap. This requires the reduction of the air and other non-condensable molecules with which the water vapor molecules will collide. An effective method is the use of a vacuum pump system.

The rate at which freeze-drying proceeds will depend largely on the rate at which the frozen object gains heat. As ice sublimes heat is absorbed; this energy is required to speed up the release of molecules in the form of water vapor. This energy is called the latent heat of sublimation. If it is not replaced the temperature of the frozen object will suffer a progressive decrease in the rate of sublimation.

For small chambers sufficient heat energy from surrounding outside air and the room temperature will suffice. The frozen object is supplied with heat input through the walls and door of the chamber through conduction or radiation.

Resistance heating, or similiar device, is normally used to supply heat energy when there are large quantities of frozen materials to undergo freeze-drying. In any case, the amount of heat energy supplied to the frozen object must not exceed the rate at which water vapor leaves the object, otherwise there can be a change from the solid to the liquid state. In such a case sublimation does not take place.

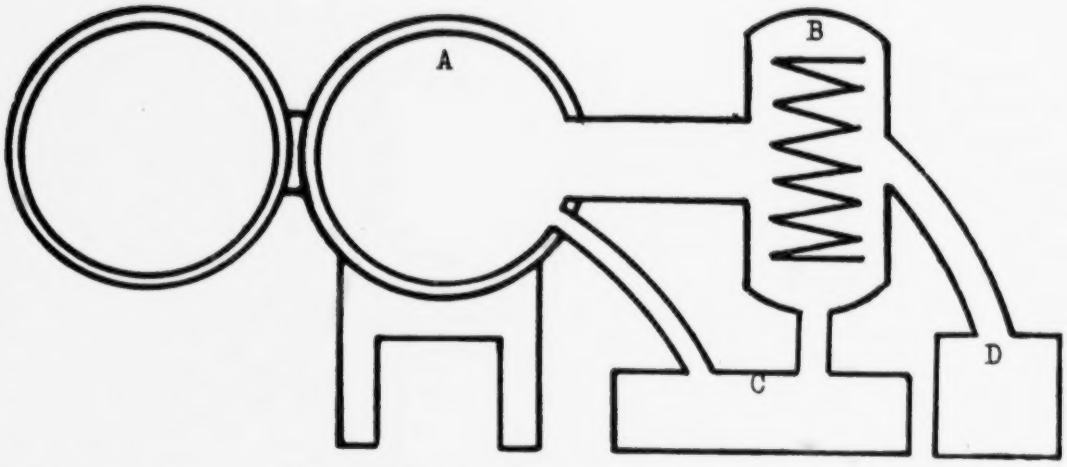

Figure 2. Simple schematic of a vacuum chamber for freeze-drying. A, chamber. B, refrigerated condenser. C, refrigeration units for chamber and condenser. D, vacuum pump.

4.3 The Basic Components of a Freeze-Dry System

Figure 2 illustrates a simplified freeze-dry system whose basic components, usually placed in series as in the diagram, comprise a chamber, a condenser, and a vacuum pump. The chamber can be of any cylindrical size as long as it can withstand an exterior pressure of 1.033 kilograms per square centimeter (14.7 pounds per square inch). It must be vacuum tight, refrigerated, and have an opening that provides easy accessibility.

The important feature of a refrigerated condenser is that it should be located in the direct path of moving water vapor molecules where they can be trapped. When contact with the condenser surface is made, the water vapors give up their heat energy, turn to ice crystals, and are removed from the system and prevented from travelling to the vacuum pump.

Condensers are primarily of two types, internal or remote. The choice depends on the application desired. In the remote type the condenser is housed in a vacuum chamber which is separate from the chamber that houses the frozen objects. This is shown in Figure 2. This type of condenser can be isolated by a valve that will permit defrosting. Smaller freeze-dryers can be defrosted with warm water or natural air. Some mid-size freeze-dryers have internal condensers designed to form an ice "plug" that can be pulled out after using hot gas to that it loose.

Apart from the technical design features required of a vacuum system, the pump should have the capacity to reduce the chamber pressure to levels below 4mm Hg. At pressures above this level the ice no longer sublimes to water vapor but turns to liquid. A look at Figure 1 will bear this observation out.

There are two refrigeration compressors in the simplified freeze-dry system shown in Figure 2. One serves the frozen object; it should have the capacity of producing controlled temperatures that go below -5°C (23°F). The other serves the refrigerated condenser and should have the capacity to produce temperatures of -40°C (-40°F) or less. Keep in mind that the force that drives the water vapor from an ice surface is the difference in vapor pressure produced by the difference in the temperature between the frozen object and the condenser.

As already pointed out, heat energy is required for the sublimation process. Where it appears that conducted or radiated heat will not suffice for the quantity of materials to be freeze-dried, heating devices can be installed. In certain types of proprietary vacuum chambers warming devices are incorporated with the design.

4.4 Degree of Vacuum Required for Freeze-Drying

For freeze-drying, Rowe and Snowman (12) state the degree of vacuum required is usually between 0.3 and 0.03 torr. These values are made up of the partial pressures of air and water vapor in the chamber, the sum of which equals the total measured pressure. For example, 0.3 torr could have an air component of 0.03 torr and a water vapor of 0.27 torr.

RHM Research Ltd (13) place frozen documents in a freeze-dry chamber (the shelves are cooled to -30°C or -22°F before loading) and apply the vacuum to a level of 0.1 torr (condenser temperature is -50°C or -58°F). About one hour later heat is applied to the chamber shelves at 40°C (-40°F). Depending on the thickness of documents the time for drying is about five days. At this stage the vacuum is about 0.06 torr.

In freeze-dry operations, the Lockheed Missiles and Space Company (14) places the frozen books on shelves and the chamber is quickly evacuated to less than 4 torr; any free water in the books is frozen. For the first 56 hours the chamber is at 10^{-3} torr (the condenser is at less than -101°C or -149°F); the shelf temperature not over 54.4°C (130°F), book temperature not over 37.8°C (100°F). When the weight of ice on the condenser is stabilized, the chamber is brought to ambient pressure, about 760 torr, it is opened, the ice removed, and if any of the books are dampish, or nearly dried, they are placed on non-heated shelves. The chamber is re-evacuated and held at 10^{-1} torr for 26 hours; shelf temperature is not over 54.4°C (130°F). When book temperatures read above 12.8°C (55°F) the cycle is completed, the chamber is brought down to ambient pressure.

5. ALTERNATE METHODS OF DRYING

5.1 Vacuum Drying

The difference between vacuum freeze-drying and vacuum-drying is, in reality, a matter of pumps. In a freeze-dry system the pump has the capacity to pull a vacuum of 4mm Hg and less; at temperatures below 0°C (32°F) water as a solid (ice) can, under certain conditions of pressure and temperature, pass to water as a vapor (sublimation). On the other hand, in vacuum-drying the pump has the capacity of pulling a vacuum of 5mm Hg and above; at temperatures greater than 0°C water as a liquid passes to water as a vapor (evaporation). A graphic representation of this phenomenon can be seen if an imaginary line is drawn in Figure 1 to represent the levels of pressures and temperatures cited.

As we have seen, the boiling point of water as a liquid can be lowered if the air pressure is decreased. The reduced boiling point at low pressure finds considerable practical application in the field of vacuum evaporation (evaporation under low pressure). This process is of primary importance in the sugar industry. Boiling off the water from the syrup at normal atmospheric pressure would char the sugar. However, the pressure is kept so low that the water may be removed at rather low temperatures.

In the aerospace industry, thermal-vacuum chambers are used to test the behavior of spacecraft such as weather satellites under simulated space conditions. Such chambers, rather large in capacity, are very effective for drying large quantities of wetted (not frozen) archival materials. In one such chamber there is a purged air system, a methodwhere vacuum pressure at 45mm Hg is applied for about one and a half hours. Then the chamber is purged with dry air of less than 1 percent humidity; the air passes over heaters at a temperature of 60°C (140°F). This operation continues for about two and a half days then the vacuum is applied again. The cycle lasts for about five days. Steam injection is used to eliminate evaporation from the wetted materials (15). Other methods of eliminating or trapping are available, for example, through the use of refrigerated condensers.

5.2 Deep Freeze Drying

There is considerable confusion over the use of terminology to describe the methods available for drying wetted archival and library materials. Therefore, at this point, it may be useful to review the terms normally used.

Vacuum freeze-drying is the process for drying frozen materials by sublimation at low pressure (high vacuum) and at temperatures below the freezing point. Possibly a less confusing term to describe vacuum freeze-drying is the single word "lyophilization." Both terms are interchangeable in the English language although the former is more in use. However,in

other languages, French and Spanish, for example, lyophilization is the preferred word; technical dictionaries give it a clear, concise meaning: rapid freezing of a material at a very low temperature followed by rapid dehydration by sublimation in a high vacuum.

Vacuum-drying is the process used to dry wetted (non-frozen) materials by evaporation in a vacuum chamber at relatively higher pressures (low vacuum) and at temperatures above the freezing point.

Deep freeze drying requires only a freezer. It is a non-vacuum process which has been described by several names: simply freeze-drying, ambient freeze-drying, cold storage drying, non-vacuum drying. The deep freeze method dries a solid by dehydration. The same laws of physics that govern sublimation apply here also. The process is quite familiar to those who have placed unwrapped food in the freezer compartment of a domestic refrigerator. Over a period of time the food becomes dehydrated; it froze, acquired a vapor pressure higher than the point of coldest temperature (freezing unit), its water vapor molecules were drawn to the freezer unit and trapped there.

Deep freeze as a method of drying is relatively well-known and has been used by a number of conservators in the past. However, the technique has not caught on perhaps because of the time required to dehydrate the frozen materials. But in a recently published article, Smith (16) describes the use of a commercial upright freezer which was modified to dry 200 to 300 wetted books in two to four weeks (and it can also be used to exterminate insects in books). The model is self-contained, has a vertical glass door freezer, and was chosen for availability of maintenance centers internationally, and its ability to cope adequately with minor disasters in libraries. The modifications include the establishment of a temperature differential between book compartment and the evaporator (freezing unit), the elimination of unnecessary heat sources, installation of temperature controls, indicating thermocouple, signal devices and timers.

5.3 Natural Freeze-Drying

Natural freeze-drying is mentioned briefly as a matter of interest and to avoid confusion with the other methods in use for drying. It is a technique for a specific purpose and is limited to those parts of the worldwhere winter climates prevail at temperatures below freezing. Note how similiar it is to the other methods already described.

Gratten and McCawley (17) write of the method; they call it "exterior freeze-drying" in which waterlogged wood is freeze-dried without the need of a vacuum chamber. They take advantage of Canadian winter: the low temperature, the daily hours of sunshine to offset the latent heat of sublimation, and the wind conditions to help remove the water-saturated air from the frozen object. A tent-like housing with polyethylene walls

protects the object undergoing treatment from undesired exterior conditions.

Conceivably, frozen documents and books could be dried under the same conditions as waterlogged wood. The process would be quite slow, but it would still be a form of freeze-drying "au naturel."

6. VACUUM FREEZE-DRYING VS VACUUM-DRYING

Conservators are often asked which of the two is the preferred method of drying wetted documents and books. At the moment there are no set answers based on standards derived from usage. And much depends on the circumstances. However, in general terms, freeze-drying appears to be the preferred method for wet books. Stabilization is immediate, the stresses a bound book would suffer are minimized, books of coated stock do not stick on drying, soluble colors and inks, fillers and adhesives stay in place.

On the other hand, vacuum-drying is a wet process. Prefreezing is not involved, it is somewhat more economical than freeze-drying, the moisture is removed by evaporation. It is widely used for wetted records. However, the advantages of stabilization are lost where danger of fungal attack exists or soluble elements are present. In this connection, Flink (18) was faced with the dilemma of drying the rare mid-nineteenth century Kleinschmidt manuscripts which were in a frozen state. Preliminary tests indicated that soluble inks and colors might feather or run with vacuum-drying. So he selected freeze-drying, by-passed the liquid phase during the drying cycle, and ended with successful results.

7. FREEZE-DRYING IS NOT A NEW PROCESS

Harris (19) notes that sublimation of ice has been known as early as the middle of the eighteenth century, but the first attempt to freeze-dry tissue was made by Richard Altman in Leipzig in 1890. His technique was to use a form of dehydration in which the tissue, previously frozen at -15°C (5°F), was placed in a dessicator, then evacuated with a vacuum pump.

During the early decades of the 1900s sublimation was little more than a series of laboratory experiments. However, the potential for industrial application of the results obtained by drying from the frozen state were apparent: the temperatures used in sublimation were low enough so that many unstable substances like those found in blood, viruses, microorganisms, other biologicals and pharmeceuticals would not undergo changes; with low temperatures there was a minimum loss of volatile substances in some foods; there was no bubbling or foaming to cause surface changes in drying; in most cases the dissolved substances remained evenly dispersed and distributed, the remaining residue highly porous with a solid framework, the solubility rapid; bacteriological growth and enzymatic changes could not take place under frozen conditions.

During the 1930s scientists and engineers saw that by establishing proper conditions of vacuum for removal of water vapor and by rapid application to the frozen product, the process could be made industrially workable (20). However, it was not until shortly before World War II that the laboratory models were made commercially available. The requirement for a vast supply of blood plasma was instrumental in the development of freeze-dry equipment and techniques. And after the war there was a virtual explosion in the use of vacuum freeze-drying for biologicals, pharmeceuticals and food. The use of this process has spread to include not only the salvage of water-damaged or wetted archival and library materials, but also of wetted textiles, saturated archaeological artefacts such as leather, cloth, wood, wrecks of old vessels sunk at sea, taxidermy, natural history exhibits for museums.

7.1 Examples of Its Use

7.1.1 Pharmeceuticals

The first time freeze-drying was used on a large scale industrial basis was during World War II for the preservation of blood plasma, the fluid that surrounds the red blood cells. The dehydrated plasma was sealed in packets and when needed was rehydrated with sterilewater. Since that time, the list has grown substantially to include medicinals that cannot be stored in liquid or frozen state: vaccines against rabies, measles, mumps, rubella; penicillin and other antibiotics; antivenin to treat snakebit; human skin cells to spread over burns; many cancer drugs such as cisplatin anddoxorubin (21).

7.1.2 Foodstuffs

Probably the product most suitable for mass production by freeze-drying is also the most familiar: instant coffee. In freeze-drying the coffee extract is frozen then introduced in the vacuum chamber where the moisture is sublimed; a solid mass is left which is then reduced to granules. These are rehydrated with hot water to make a cup of coffee. Other products include meats, vegetables, and fruits. Some fruits like strawberries lend themselves superbly to freeze-drying: one kilogram of freeze-dried pieces gives ten kilograms of strawberries after rehydration (22). Other products that lend themselves to the process include spices, dairy products, and food packs.

7.1.3 Biological Specimans

Examples of biological specimens which are freeze-dried in laboratories on a routine vasis are bacterial or virus suspensions, ensymes, fungi, protozoa, tissue, bone and skin for surgical grafting (12). In the macrobiological area museums and taxidermists freeze-dry exhibit specimens such as rodents, reptiles, birds, crustaceans, insects and fishes. In freeze-drying of these specimens there is faithful retention of appearance and tissue structure - prevention of shrinkage - which is vitally important. Animals as large as a six foot alligator

have been successfully freeze-dried; apparently the only limitation to the size of the animals is the capacity of the vacuum chamber in which they are dehydrated (23).

7.1.4 Archaeological Artifacts

When archaeologists find ancient artifacts of wood, cloth or leather which have been in wet or damp sites, they face the immediate problem of drying the object without irreversable shrinkage, surface checking or other deformations. Freeze-drying is one of the methods employed successfully in drying pre-treated (polyethylene glucol impregnation) wood in order to prevent the normal movement of water which would cause the collapse of damaged cells (24). Leather items have been successfully preserved by using a similiar technique (25). And neolithic textiles along with braided work have been salvaged by freeze-drying after thorough cleaning and desalinization (26).

8. EARLY EXPERIMENTS IN FREEZE-DRYING BOOKS AND DOCUMENTS

8.1 Precursors

8.1.1 Canadian Entities

One of the first known reports on attempts to use freeze-drying for the salvage of water-damaged library materials was published after the Library of Parliament fire in Ottawa in August 1952. In order to extinguish the blaze sprinklers were kept on for six hours. As a result thousands of books were soaked or moistened. Experiments on drying were carried out by the Low Temperature Laboratory of the National Research Council and in the Forest Products Laboratories of the Department of Resources and Development. Included among the methods tried were freeze-drying and vacuum-drying. The reported difficulty with these two methods was the slowness and relative inefficiency of attempting to dry the soaked books from the outside inwards (27).

8.1.2 Smithsonian Institution, Washington, D.C.

Hower (23) of the Smithsonian Institution published a report on the freeze-dry preservation of biological specimans for exhibitions in museums. He describes a new application of the freeze-dry technique carried out in the 1960s after a copy of "Merchant's Almanac" was retrieved from a river steamer sunk in the Missouri River in 1865. The almanac had been submerged for over 100 years. It had been placed in a commercial deep freezer at about -20°C (-4°F). Dr R.M. Organ, Chief of the Smithsonian's Conservation Analytical Laboratory, asked Dr Hower to experimant with part of the almanac to see if vacuum freeze-drying would work. The specimen was placed in a large vacuum chamber at -20°C and the system pumped down to 150 microns (0.15mm Hg). The book was removed before it was totally dry in order that it have sufficient moisture to remain flexible. The initial weight of the specimen was 952.9

grams and final weight 522.9 grams. In the report Dr Hower expresses his interest to work with more material of this type in the hope of establishing standard procedures.

8.1.3 Technical University of Denmark

Probably the first attempt to vacuum freeze-dry water-damaged library materials with marked success was one carried out by Flink and Hoyer (18) in 1971. It all started during the winter of 1968 when a fire was being extinguished in the Greenland Regional Library of Godthab on the west coast of Greenland. Because of the weather conditions prevailing at the time, water from the fire hoses froze the water-soaked materials housed in the library. The most valuable items, packed in cardboard document cases, were hand-written letters, manuscripts and maps which belonged to a missionary of the mid-nineteenth century, Samuel Kleinschmidt (18). The frozen items were transported in that state to Copenhagen where they were kept in cold storage in order to have time to find the best course of action for the drying process.

The problem facing the technicians in Copenhagen was that the ink used in the documents would run if the conventional air-drying technique were to be used for thawing. The frozen books posed no problem and were air-dried. But it was determined that this technique was too risky for the manuscripts. The decision was made to use freeze-drying.

The necessity for freeze-drying the documents was demonstrated by an experiment in which paper marked with various modern writing materials (ink, ballpoint pen, felt tip marker) were immersed in water, frozen, and then either air or freeze-dried. The freeze-dried samples were perfect, whereas the writing on the air-dried samples had run.

Twenty-seven storage packets and a photograph album were delivered to the Food and Technology Laboratory of the Technical University of Denmark, Lyngby, where the freeze-dry chamber (Atlas Ray 1) was located. The items were held at -30°C (-22°F) until the start of the freeze-dry operation. By careful manipulation the frozen packets were removed in order to eliminate a large resistance to mass transfer while undergoing freeze-drying.

To improve the rate of drying (for convenience rather than necessity) and to ensure sufficient clearance between the chamber's heater shelves, some of the thicker stacks of frozen documents were pried apart while frozen. This was possible without damage to the manuscripts only because paper folders had been used as separators within the library holder.

Stacks of frozen papers, two to three centimeters (about 3/4 to 1 1/14 inches) thick were placed on pre-cooled, porous trays and conservation freeze-drying conditions were chosen so that no melting or color change would occur. Radiant heating at a plate temperature of 45°C (113°F) and a chamber

pressure of 200 mtorr (0.2mm Hg) was used. The weight of the sample was monitered by using the balance built into the freeze-dryer; drying was continued to constant weight. The average drying time was about one and a half to two days. When drying was complete the chamber vacuum was slowly released (18).

Flink summarized the outcome of the operation as follows: The freeze-dry process gave perfect results as each page separated easily, and in no case did the ink run. Manuscripts which had been compressed and misshapened before freezing retained that shape after drying. These samples were later satisfactorily treated in the usual manner of pressing and binding. The photograph album (from 1906), freeze-dried at the same time, presented some problems. Prints which were frozen emulsion-to-emulsion often stuck together after drying. Soaking the stuck prints in water and then carefully separating under water usually solved this problem. Due to the thickness of the album (ten centimeters; about four inches), freeze-drying required four and a half days. Even with this length of time six photographs at the center of the album were still frozen and melted before they could be reins ted into the freeze-dryer, ruining the handwritten captions. This indicated that freeze-drying had indeed been necessary to conserve successfully the handwritten documents (28).

9. THE USE OF VACUUM CHAMBERS FOR RECOVERY OF WATER-DAMAGED ARCHIVAL AND LIBRARY MATERIALS

9.1 Case Histories

Apart from actual experience the best way to acquire familiarization with the subject is to see what others who have suffered a disaster have done. Some of the case histories that follow may serve the purpose even though at first glance they cannot be called typical: thousands of documents and thousands of books in large institutions are involved. However, the basic difference between the overwhelming task of salvaging 50,000 books or 450,000 archival files, and a small disaster that wets a few books and documents is one of magnitude. The same steps taken by a large institution for recovery operations apply to a small, modest one as well.

9.1.1 Corning Museum of Glass Library Flood, Corning, New York, June 22, 1972.

On June 22, 1972, the Corning Museum of Glass was submerged by flood waters as a result of a hurricane that swept the area. There were three days of continous, heavy rainfall. A normally shallow river that flows through the city crested to nearly 30 feet (9.14 meters); the nearby Corning Museum had its main floor inundated to a level of five feet or one and a half meters (29).

Apart from the damage to a number of highly valuable objects in the glass collection, the library, which housed one of the best collections on the art, history and archaeology of glass, was badly damaged by the flood waters.

Everything printed on the subject of glass had been collected for years and included books (a large number of them printed on coated paper), periodicals, documents, pamphlets, prints, catalogs, manuscripts, incunabula, medieval manuscripts and other rare books.

On the morning of June 24 the sight that greeted museum personnel who entered the library was the devastation of books so sodden and swelled that the sides of the stacks were bent. Crowbars had to be used to dislodge the books. Other shelves had popped volumes into the muddy water; incunabula, manuscripts and rare books were submerged in it.

The difficulty of know ng what to do and where to start was eased by librarians who had read articles on the freezing of water-damaged books in order to stabilize them. But the obstacle of the moment was that electric power was out in the city. Furthermore, with so much damage to the community there was no gasoline available, no commercial transportation, no running water. And all were aware that if salvage operations did not begin immediately, mold infection would take place. So, with the librarians directing, members of the staff and volunteers began to gather the damaged materials.

Very little cleaning could be done without clear water. The books were wrapped in paper towels to hold them together and to separate one from the other. Plastic material would have been better but none was available. Then, using whatever containers on hand, the volunteers delivered the materials in their own vehicles to areas that did have electricity and placed them in domestic freezers. About 30 miles (40 kilometers) away cold storage facilities were found and used.

It took five days to pack the damaged collection of approximately 6,500 books, pamphlets, file catalogs, and other materials. Some 30 boxes of office files were packed for freezing as well. The card catalog had been ruined by water and thrown away. Fortunately, the shelf list although damaged was usable. The undamaged books were stored in a nearby college. By the time the last volumes had been packed and taken to storage freezers mold began to appear.

In August the undamaged books had to be removed from the college since the fall semester was about to start. A place was found in the city - an empty supermarket - where a library/restoration studio was established. Also, the scattered frozen materials were placed in a rented freezer-truck which was parked next to the makeshift library/restoration studio.

A program charting the course to pursue was established which included the decision to send 200 rare books, in their frozen state, to a professional restorer. A chemist was also hired to supervise the work of volunteer workers. The chemist was also authorized to use new techniques to cut the time it would take for restoration of the damaged materials.

It was soon found that the freezer truck was so crammed there was no room for grouping and sorting the frozen materials. Also, the freezing components of the truck acted erratically causing partial thawing of its contents. The problem was solved by obtaining two large walk-in freezers. As each item was moved into the new freezers a record with copies was made of its condition. One copy remained with the item, one copy to the conservator, another to the librarian. When the item was taken out from the freezer the conservator noted the record which had the librarian's judgement on how to restore: restore fully by traditional methods, by new methods, or hold for later decision.

Thanks to a foundation grant a research scientist was hired for technical advice. The scientist and the conservator began to investigate drying methods. They focused on three:

a. Thaw the books naturally. (Pull the binding, wash to remove mud and stains, deacidify, dry, collate and store until the pages could be microfilmed).

b. Dry with a commercial dielectric drier. (The frozen book is placed between two electrode plates and electrical energy is shot through it. But it was soon found that the wet covers had to be removed because they made drying of the block difficult. All metal objects, clips and staples, had to be removed to prevent arcing. Otherwise, the paper could be charred and burned. Since it was unknown what damage was being done to the paper, the valuable books were only thawed by this method but not dried).

c. Vacuum freeze-dry. (This technique was considered even though for the situation at hand it did not seem very practical. There were no vacuum chambers in the vicinity; it was not known what would happen if this technique was used on the large proportion of books printed on coated stock).

By the time the first year ended about 3000 books and boxes of files had been dried, cleaned, and made ready for microfilming. Also, the staff scientist had researched the chemistry of coated stock and run tests on wet books printed on coated paper in order to find why such books turn to solid blocks on drying. The scientist also established the parameters which could be used in the freeze-dry process (30).

It was decided to use the vacuum chambers located at Valley Forge, Pennsylvania, operated by the General Electric Company. Using the parameters established by the staff scientist for coated stock, the remaining frozen volumes were subjected to a process that took nine days to complete. At the end of that time 125 books had a condition that ranged from damp to wet. But 95 percent of the coated stock was saved, possibly marking the first time such success had been achieved.

The five percent loss was due to text block solidification before the books had been put in the chamber for freeze-drying.

By December 1974 the restoration mission was over; some 6500 books had been dried and cleaned together with 30 cartons of documents and files.

9.1.2 Charles Klein Library Fire, Philadelphia, Pennsylvania, July 25, 1972.

The Charles Klein Law Library, located on the campus of Temple University in Philadelphia, was built originally in 1891 as a synagogue. The three story and basement building was taken over in 1953 by the University and renovated for use as a law library. The library held a 400,000 volume collection that included rare books on English and American law and documents that belonged to Benjamin Franklin. On July 25, 1972, a fire broke out on the third floor of the building, the cause was later thought to be of electrical origin, which resulted in its destruction along with a heavy loss of the library collection. The building had water-type extinguishers. There was no automatic sprinkler system installed; the manual fire alarm system was for alerting personnel in the building.

On that hot and humid day, at 1:45 pm, the Philadelphia Fire Department received a telephone alarm which resulted in the dispatch of four engines and two ladder companies to the Charles Klein Library which, at the time, was billowing smoke from the roof. The firemen first on the scene extinguished visible fire on the third floor but heavy smoke continued to develop in the area. A second alarm was sounded, then a third at 2:30 pm.

While the fire companies were busy on the third floor, a group of professors, students, and onlookers formed a human chain to salvage books shelved on the main floor. In this manner, the card catalog and about 3,000 volumes were saved.

The fire authorities halted the impromtu salvage operation when it became evident that the ceiling of the main floor was in danger of collapsing. At about 2:35 pm, not only the ceiling but the roof collapsed. Two minutes later the fourth alarm was sounded.

By now, master streams from ladder pipes and elevated platform apparatus were placed in service along with hand lines to contain fire. The combination was pouring 11,000 gallons (50,000 liters) of water into the burning building (31). A fifth alarm brought additional firefighters to the scene to replace those exhausted by the hot, humid weather. The total contingent was now 20 engine companies and one water supply company. At 3:15 pm, an hour and a half after the first alarm, the fire was declared under control.

The preliminary damage assessment, terse and concise, was as follows: Water stands three feet deep (about one meter) in the basement stacks and rare book room; main reading room books

badly damaged, those toward the center burned when the roof fell in, others soaked with water from hose streams; some shelves and the entire Lucas Hirst collection of 21,000 books in a separate room are preserved by salvage covers placed over them early in the fire (31).

The company that carried insurance for the University obtained the services of two conservators from the Preservation Office of the Library of Congress, and one from the Philosophical Library in Philadelphia. Up to this point no general salvage plans had been made. The first objective was to enter the basement, check the damage, and stabilize mold damage to the wet volumes. The high humidity and temperature found in the basement required prompt action. Thus, these volumes were given first priority.

On Friday afternoon, July 28th, emergency lighting and fans were set up in the basement to provide adequate working conditions. By midday, Saturday, volunteer crews began salvage operations. During the weekend some 30,000 volumes were removed and put in cold storage at 20°F below zero (-30°C). The majority of these volumes were from the three bottom shelves where the most water damage had occurred. Among the moldy, wet, and distorted books were many irreplaceable documents.

The volumes were wrapped in freezer paper and placed in boxes, loaded on four freezer vans, and transported to the cold storage facility. The books were not catalogued before packing. It was determined that at some later date the library staff could remove the books from cold storage and decide what course of action to take. The remaining volumes in the basement were given eight sprayings of thymol to retard mold growth (31).

On Thursday of the following week the remaining 120,000 volumes in the basement were removed by volunteer crews. The books were indexed, boxed, and loaded on trucks. As mold growth had been stabilized with thymol, these books were transported to a controlled atmosphere facility (31). Thus, the stability of all the materials salvaged from the disaster was assured.

It was decided to freeze-dry the frozen materials. The insurance carrier, through its subsidiary salvage company which had arranged for the cold storage facilities, boxes and transportation after the fire, contacted the General Electric Space Simulation facilities at Valley Forge, Pennsylvania, for the possible use of a space chamber. The appropriate arrangements were made and technicians at the space facility made some preliminary experiments in a laboratory chamber to determine the conditions that would be required for the frozen books to reach a dry state.

In order to freeze-dry the frozen books as economically as possible, a chamber was selected that would permit large

volume drying. The chamber had a capability for easy horizontal loading and permitted an arrangement of shelves to be set up for the books. It was decided that these would be located spine down for better surface evaporation and closer contact with mesh heaters used to supply heat lost by sublimation. The chamber was equipped with a condenser that used liquid nitrogen as the refrigerant to trap the water vapor from the frozen books.

The processing took about five days including loading, unloading the shelves, and packing for shipment. For two of these days the books were in the vacuum chamber. About 3,500 books were treated at one time. Shoulberg (32), Applications Engineer at the facilities, estimated that in the first batch 80 percent were dry; the second batch yielded 97 percent. The books that were not thoroughly dry in one batch were included in the next load.

During the process some experiments were carried out in the vacuum chamber by stacking the books spine down in boxes, and also putting the boxes directly on the shelves. Books in open boxes appeared to dry as well as those not boxed. An observation was made that if freezing is required and properly sized boxes are available, the boxes can be stacked spine down with paper in between and frozen in the boxes. The books can be dried directly in the type of chamber used at the facility with a great reduction in handling (32).

One final observation that relates to the hazards posed by water: In reference to the Charles Klein Law Library fire, E. Wiley of the National Fire Protection Association said that "The fire could have been controlled by an automatic sprinkler system so arranged as to transmit an alarm to a central station or to the fire department. Sprinkler flow would certainly have caused less damage than fire department hose streams." (31).

9.1.3 National Personnel Records Fire, St Louis, Missouri, July 12, 1973.

The National Personnel Records Center is a specialized organization that holds, for the most part, the service records of personnel, both military and civilian, who have been in the federal government. The records are housed in a six story building made of concrete with exterior walls of aluminum and glass. The building, which stands on a large tract of land, was completed in 1956 after a team of experts spent several months studying the spatial needs and facilities required to perform the task of a records center. However, the building had no sprinkler system. It appears that before the construction began the architect's surveyteam sought the opinion of government officials on the installation of a sprinkler system. Opinion was divided. However, the day was won by those who felt that damage from water was a greater threat than that from fire. The system was not installed (2).

On July 12, 1973, a few minutes after midnight, a fire of unknown origin broke out on the sixth floor of the building, In danger of destruction were the Center's 120,000,000 personnel files stored in cardboard file boxes and file cabinets distributed throughout the building. The response of the fire companies was rapid. However, conditions were such that alarm after alarm went out and, eventually, firefighting units from 42 districts were on the scene (2).

Initial efforts to fight the fire consisted of a combination of snorkels pouring water from the outside, and hose companies doing the same from the inside. But conditions became so bad that the internal efforts were abandoned. The threat of a general spread of fire to the fifth floor was averted through the continuous flood of water to the sixth floor during July 13. By early morning of the next day, firemen were on the sixth floor. On the afternoon of July 15, heavy smoke began pouring from the southwest corner of the building. Firemen were dispatched up a ladder to direct more water there. By the morning of July 16 the crisis was over. Only one company of firemen remained. But the long days of recovery and rehabilitation were just beginning (2).

Waters (15), who was an adviser at the scene of the disaster, gives a graphic description of the amount of water used and extended over the area: There was something like 60,000 gallons (272,400 liters) perminute on the fire, and a total of just under 100,000,000 gallons (454,600,000 liters) on the whole area. For a total of three weeks after, there was water on every floor at a depth of one to one and half inches (about 25 to 38 millimeters) at a weight of 8,000,000 pounds (3,628,800 kilograms) per floor. The drainage that was possible through these floors came down elevator shafts, escalators, and water hydrants.

Stender and Walker (2) estimate that only ten percent of the 22 million personnel jackets stored on the sixth floor at the time of the fire could be recovered. As to the lowerfloors, more than 10,000 cubic feet (280 cubic meters) of water-damaged records were removed. Waters (15) estimated that about 450,000 files were damaged below the sixth floor.

In any event, officials from the parent organization in Washington, the National Archives and Records Service, found themselves facing the largest records drying operation ever undertaken (2).

One of the first problems that had to be faced, in light of the tremendous amount of dampness in the building and the high summer temperatures, was attack by mold. The Preservation Office of the Library of Congress recommended spraying with thymol in solution.

In the meantime, temporary facilities were found at a nearby civilian records center where hastily constructed drying racks were assembled for the classid method of drying by

exposure to air. As the damp records were assembled for transfer by truck, they were sprayed with thymol. It was found that the plastic baskets used for transporting milk containers were ideal for open-shelf drying. Soon an initial small supply of these baskets grew to some 80,000. Eventually all the storage areas were sprayed by a local exterminating company (33).

Some days after the fire, officials from the National Archives and Records Service learned of the existance of a vacuum chamber facility at the McDonnell Douglas Aircraft Corporation in St Louis used for the national space program. The chamber was designed to simulate conditions in outer space during the ground testing of space systems used in the Apollo series of space shots (2). It was decided to approach the aircraft officials and ask for permission to try their vacuum chamber for drying the wet records.

With approval granted, some hurried experiments were undertaken to see what would result with this particular type of drying operation. As soon as the initial tests were completed satisfactorily the drying of records proceeded on a priority basis. The now familiar plastic baskets were packed three quarters full in order to leave air space. Since they can be interlocked when placed one atop the other, they were packed on pallets and loaded on trucks for shipment to the vacuum-dry chamber. About 60,000 to 70,000 cubic feet (1680 to 1960 cubic meters) of materials were scheduled for drying in a chamber with 2,000 cubic feet (56 cubic meters) capacity. At this rate it would have taken several months to complete the task. However, use of the first chamber at the McDonnell Douglas Aircraft Corporation was supplemented eventually by the use of two additional chambers and, finally, by a similiar chamber at a National Aeronautics and Space Administration facility at Sandusky, Ohio (2).

The vacuum-dry system was very effective. During a typical loading of one chamber holding 2,000 baskets about eight pounds (3.6 kilograms)of water were removed from each container. Thus, a total of nearly eight tons (7200 kilograms) of water was removed during each loading (2). Freeze-drying was initially considered a possible course of action for drying by sublimation; temporary arrangements had been made to place railroad freight cars on a siding near the records center in case they were needed to freeze and stabilize the water-damaged records. However, as things turned out, the nearby chamber facilities and success of the operation made the use of the freight cars unecessary.

9.1.4 The Stanford Meyer Library Flood, Stanford, California, November 4, 1978.

The Stanford Meyer Library, part of the Stanford Univercity complex, is located in the basement of the J. Henry Meyer Undergraduate Library. The basement library houses 400,000 volumes which are used by undergraduates for research in social science and humanities. The book stacks are on two levels of a metal-tiered construction.

On November 4, 1978, at 2:51 am on a Saturday, an eight inch (20 centimeters) water main burst at a point some 20 feet (about six meters) from the nearest wall of the Meyer building. Water from a crack along the main filled a large excavation, part of construction work in progress, and then found its way through a number of holes in the building wall which had been drilled for the installation of drains and pipes.

Water entered the building and flooded the floor level of the upper metal-tiered stack which had a surface of 5,000 square feet (460 square meters). From here the flood water dripped to the floor below and soaked, to varying degrees, more than 50,000 volumes. Thanks to an emergency call to a plumbing crew working on the campus, the water was turned off 20 minutes after rupture of the main.

As the water travelled the short distance to the basement wall, it carried with it fine silt which was deposited over the floor on both metal-tiered levels. Many books had swelled to the point where they were jammed into the shelves; others had popped onto the silt-covered floor. There were never more than a few inches of water standing on the two floors, However, the water dripping like heavy rain for several hours on the lower level did most of the damage.

The building's electric power was kept on in order to start cleaning operations with sponges, mops, and large vacuum cleaners designed to operate in wet or dry conditions. Most of the silt and water were removed by 8:00 am, the same day of the disaster.

An initial meeting was held at 6:00 am to carry out certain steps already developed by Stanford's disaster planning team. The steps included the appointment of a disaster coordinator, the maintenance of the building air-handling system to maximize the circulation of cool air to reduce humidity as quickly as possible, and to freeze the materials right away (4).

Cold storage facilities were located a short distance from the University. Some 3,000 books cartons and milk crates were initially ordered. But 1,500 more were obtained because the books, wrapped in freezer paper, were packed one layer deep and spine down to avoid damage. Cartons were packed three high to avoid collapse of the damp boxes; 160 pallets used to carry the boxes were sent to the freezer. The boxes remained on the pallets until they were removed at the vacuum chamber at a later stage.

By 9:30 pm, Sunday, all materials had been removed to the freezer. Forty-three hours had elapsed from the first alarm until the final freezing. There was no evidence of mold. At -20°F (-28.9°C) the wet materials were stabilized and safe until decisions on drying techniques could be made (4).

The literature was reviewed with particular attention to past disasters, for example the National Personnel Records Center fire, where vacuum-drying had been used for mass drying of water-damaged documents. It was evident to University officials that for the frozen books, freeze-drying was the best method to employ. They knew that machines for this specialized drying are used in scientific laboratories, food processing, and in the aerospace industry. So, a search began. Several were located which might have been suitable for a small collection of water-damaged materials, but not for over 50,000 books.

Finally, a facility was found at the Lockheed Missiles and Space Company at Sunnyvale, near the cold storage freezer where the books reposed. Several vacuum chambers were possible; the one selected had walk-in accessibility and sufficiently large, 18 x 18 x 36 feet (5.5 x 5.5 x 10.9 meters), to take metal racks capable of holding 5,000 frozen books with provision for a surface heating system. The vacuum chamber had a ten by ten foot (3.05 by 3.05 meter) condensing panel, cooled by liquid nitrogen, to trap the water vapor from the frozen books.

A trail run was made to determine the best method for housing the books: in milk crates, or books upright in their normal position, flat on the shelves, or simply in boxes. The books selected - discarded ones - were soaked in water then frozen. They were placed in the chamber to freeze-dry. When the drying cycle was completed the dried books turned out to be brittle - less than two percent moisture. Ideal moisture content is between five to seven percent (33). The books would require stabilization in a normal environment.

The test also proved that the manner of housing made no significant difference, but wrapped books dried more slowly than partially wrapped books. It was also found that books placed upright looked better physically. So, a decision was made to dry the books upright, spines to the back, supported snugly by size (4). Heat would be applied to the spines, and thermocouples placed in some of the books to record the drying state.

On February 5th the first load, 5,000 frozen books, was transported to the chamber and placed on the shelves. It was estimated that about four days would be required for all phases of the drying operation.

The first step was to evacuate the chamber to less than 4 torr to freeze any free water in the books. Then for the first 56 hours the condenser panel was to be kept at less than -150°F (-101°C) with the vacuum in the 10^{-3} torr range. The book temperature would not exceed 100°F (37.78°C). At the end of this period the power required to maintain these conditions would be turned off and the chamber brought to normal pressure. Ice from the condenser would be removed and books checked for dryness. Those found to be dry would be placed in a non-heat area of the shelves. Next, the chamber would be re-evacuated to a pressure of 10^{-1} torr for about 26 hours with the book

temperature at 100°F. This stage would terminate when about one half of the thermocouples read at temperatures above 55°F (12.78°C). The chamber would then be brought to normal pressure. Any books still frozen would be placed with the next load to dry (14).

With much learning on-the-job, by February 12th some 10,000 books a day, two days a week, were being dried by sublimation. Care had to be exercised with the dried volumes because of their fragility in that state. March 12th was the date of the last load to leave the chamber. Most of the books were placed on shelves in a vacated school site near the University. The conditions established there enabled the books to stabilize to six percent moisture content,as measured by an Aquaboy (35), in about five weeks after shelving.

By the end of the year the operation was closed. Much work had been done since the flooding of over 50,000 volumes: besides freezing and drying, the cleanup of the flooded library, repairing and restoration of books, among other details. One thousand books were bound, 27 items were microfilmed, 12 volumes would require searching for replacement, 34 books were discarded due to the flood, three were not replaceable. All other items were returned to the stacks (4).

9.1.5 Taylor Institution Library Flood, Oxford University, January 1979

During the month of January 1979 a water main burst at the Taylor Institution Library and flooded the basement stacks where approximately 2,000 books, some rare volumes from a collection of Slavonic literature which included early 16th century examples, were water-damaged in degrees varying from damp to very wet.

A small number of slightly damp books were left on the shelves to dry. The moderately wet, about 1800, were dried in the classical manner by standing them upright and slightly open in a current of dry air. The remainder, too wet to stand upright, were put in plastic envelopes, frozen, and kept in a commercial chest-type freezer at -18°C (-0.4°F). There was now time to seek a safer and quicker method for their drying.

Laboratory scientists at the Atomic Energy Research Establishment, at Hartwell, where industrial drying was being researched, were approached with the problem. It was decided to adapt a vacuum chamber, 48 inches in diameter and 56 inches long (122 centimeters in diameter and 142 centimeters long) to do the job. Shelves were built into the chamber on which the frozen books could be placed. The books varied in size and material: rag paper to wood pulp, covers that ranged from vellum to leather, some with paper or plastic covers on ordinary boards. The books contained from 35 percent to 100 percent of absorbed water as determined by weighing during the drying process and compared with the final weight of each book (36).

Initial trials were undertaken using a small quantity of frozen books chosen at random; vacuum pressures were varied, different amounts of silica gel in trays were used to absorb the water vapor in the chamber. Finally, the conditions for the production runs were: A vacuum pressure of 745mm Hg in the presence of two trays each with four kilograms (8.8 pounds) of silica gel which was renewed each 24 hours. Ten grams of thymol crystals in a dish were used to prevent mold attack. It was found that small books reached a dry state after 80 hours, the larger after 225 hours (36). As the books were removed from the chamber, they were placed in a normal atmosphere for about 48 hours to stabilize them.

The condition of the dried books varied. There was light wrinkling of the pages with some staining due to metallic salts in the flood water. There was damage to the spines, hinges and joints caused by the initial soaking. The vellum and leather covers were affected by the drying: The vellum became brittle when dried out; the leather suffered where stretched over warped boards. The more modern materials used for covers recovered well, except where plastic materials were used. These warped badly on drying.

Of the 115 books sent to Harwell for drying, 43 needed to be rebound (36). It must be emphasized, however, that materials requiring rehabilitation after vacuum drying were already damaged at the time of freezing and drying.

9.1.6 Regional Office of Income Security Services Fire, Winnipeg, Canada, January 22, 1981.

On January 22, 1981, a fire gutted the three story building in Winnipeg that housed, on the upper floors, a regional office of Income Security Services. Also, in the same building, there was a second social service organization: The Jewish Child and Family Service. The two services had up to 600 linear feet (183 linear meters)of records. During the fire about 80 file storage cabinets went through the floors as the firemen battled the blaze.

The salvage operation began when the active files of the Income Security Services were moved to the basement of another building. About ten percent of the files were relatively unharmed, but a large amount of file cabinets had swelled with the water; crowbars had to be used to pry them open.

Mold infection and the fusion of wet documents became the immediate concern of the archival personnel. So, it was decided to air-dry on the spot. Heat was reduced, work tables were set up, new materials for storing the dried documents were obtained. Large fans were placed in strategic locations to blow air over the documents. With relatively dry air in the basement, about 30 percent relative humidity, the manual drying process, aided by hand-held hair dryers, progressed satisfactorily.

Attention was now turned to the closed but wet files in some 150 file drawers. Plastic milk crates were obtained from a local dairy. It took over 400 to hold about one cubic foot (0.028 cubic meters) of loosely packed documents in each crate. On January 29th the crates, which had been stored in a cool place, were moved into a building with no heat. In a city where winter temperatures range from -7°C to -28°C (19.4°F to -18.4°F) the wet documents were effectively frozen.

Locating a facility for the drying operation was the next problem to solve. A telephone lead provided the required information: The Canadian Forces Base at Winnipeg had a high altitude vacuum chamber. Soon the archives authorities were in touch with the staff of the School of Aeromedical Training. It was discovered that members of the staff had experience in drying wet documents in such chambers. At the same time, experts at the Library of Congress were contacted for advice.

The Canadian Forces chamber is 20 years old with an interior that measures 24 feet long by 8 feet wide and 6 1/2 feet high (7.32 meters long by 2.44 meters wide and 2.0 meters high) (37). The chamber was of sufficient capacity to permit the entire load of frozen documents in the crates into it. This phase was completed on February 3rd.

The chamber lacked a heat source to replace the heat lost during evaporation. As a consequence, the drying process was slow. After four days the vacuum chamber, which had been at a simulated altitude of some 80,000 feet (24,400 meters), was reduced to normal pressure to run a check on the documents. The process was working, but they were quite damp. So, the drying cycle was repeated. Three days later over half of the documents were dry.

The remaining damp documents were distributed among additional crates in order to facilitate drying; the load went back into the chamber and its simulated altitude. On February 12th, after nine days under vacuum pressure, the chamber was brought down to normal pressure. The small amount of slightly damp documents were easily air-dried with the help of fans.

10. OTHER VACUUM AND FREEZE-DRYING ACTIVITIES

10.1 A Selection by Countries

The use of freezing as a method of stabilizing water-damaged archival and library materials, followed by vacuum-drying or freeze-drying, is gaining more and more acceptance internationally. The purpose of the following country-by-country sampling is to give the reader an idea to what extent this is true.

10.1.1 Austria

April 1985. At the Institute for Restoration, Austrian National Library, Vienna, freeze-drying techniques have been carried out in an old, non-controllable installation where water-soaked books are placed for drying. The process takes more than four weeks. However, there are plans underway to install a modern freeze-drying chamber which will dry such materials in about three days. The chamber will also be used to size and strengthen newspapers; bound newspapers will be impregnated with a solution that strengthens and deacidifies (38).

10.1.2 Canada

February 1977. On February 11th, the building that houses the Engineering Library at the University of Toronto was destroyed by fire. Water was poured into the building for over seven hours. The cold weather produced one benefit: the water-soaked books were quickly frozen and stabilized. Some 500 of these books, considered rare or difficult to replace, were put in the high altitude chambers of the Canadian Forces Base at Downsview, Ontario, in accordance with the University's Disaster Contingency Plan (39).

10.1.3 England

During 1981. A scientist of the Lord Rank Centre for Research, High Wycombe, and a conservator from the Bodleian Library were discussing accelerated freeze-drying used to preserve foodstuffs. They agreed that the process could be used to dry water-damaged books. When Lord Vernon's collection of valuable 16th and 17th century books at Sudbury Hall, Derby, was damaged by a flood a number was taken to the Centre, quickly frozen, and the ice sublimed away in high vacuum. There was no damage to paper or bindings (40).

Reported in 1985. A.P. Bush (41), Archive Conservation Section, National Maritime Museum, reports an experiment in freeze-drying books in a commercial freezer (non-vacuum technique). A set of nearly identical books (wove paper, case bound, cloth, strawboards) was soaked in water and placed in a commercial cold storage facility. Eight of the books were soaked for 18 hours, taking up to about 50 percent of their dry weight in water. Seven were frozen at $-25^{\circ}C$ ($-13^{\circ}F$). After 16 weeks three books which had been wrapped in plastic film lost two percent of their water; three which had been stacked together and wrapped in paper lost 33 percent; one book wrapped in paper and placed separately for maximum exposure lost 75 percent of its water. After 51 weeks the books were removed; none was totally dry. The books not wrapped in plastic had lost about 81 percent of their water.

10.1.4 France

Reported in 1985. In 1981 a petroleum platform, the Alexander Kielland, sank in the North Sea. Three years later, the platform was put afloat and numerous salvaged documents were put in plastic bags and frozen at -20°C (-4°F). The documents, which hopefully would supply clues to the cause of the disaster, were varied. Most were like solid blocks when recoverd and included books, photocopies, paper registers, and carbon paper. The texts were either printed, written in ink, pencil or ballpoint pen. The frozen materials were taken to USIFROID, Maurepas, France, where a vacuum chamber sublimated the ice through an elevated temperature. The materials recovered their flexibility satisfactorily; the centers of the books were in a perfect state of conservation. The colored inks on the rolls of register paper had not degraded despite their degree of solubility; pencil notes on paper were perfectly preserved (42).

10.1.5 Germany (FRG)

Reported in 1972. Eichhorn (43) reports the use of "deep freezing" for the stabilization of soaked books. After a flood in Stuttgart that damaged thousands of books stored in library cellars, the materials were placed in industrial cold storage for freezing.

Reported in 1973. Elmer (26) describes the freeze-drying of Neolithic textiles and braided work. These artifacts were first thoroughly cleaned and desalinated, then dried in a vacuum of 1 to 0.5 torr at temperatures of-17° to -24°C (1.4 to -11.2°F).

Reported in 1973. Eichhorn (44) writes on the use of deep freezing for the storage of water-soaked books. These are stored at -20° to -30°C (-4° to -22°F). The intention is to thaw them slowly until dry. Eichhorn notes that it is possible to dry books in the frozen state with a vacuum chamber.

Reported in 1976. The Institute of Space Simulation, Federal Center for Aeronautic and Astronautic Investigation and Testing (Cologne) has found that space chambers can be used successfully in recovering books, manuscripts and documents which have suffered deterioration as a result of high environmental humidity, broken pipes, or floods. Small chambers are best in which the principle of physics is used where water evaporates with greater speed as the pressure is reduced. A temperature of 50°C (122°F) is maintained in the chamber while the interior pressure is kept at 5 and 10mm Hg by the vacuum pumps. The extracted humidity is transformed into ice on special freezers. In experiments carried out with wetted paper, its dehumidification takes from 20 to 40 hours depending on the quantity. Deterioration is minimal. Wet books with pages stuck together can be used without difficulty at the end of 24 hours. The cost of running a thermal vacuum chamber with a volume of three cubic meters

(106 cubic feet) for 24 hours is 300 marks. A smaller chamber with a volume of one cubic foot (0.028 cubic meters), into which 250 kilograms (551 pounds) of paper can be placed for treatment, costs one half the amount (45).

Reported in 1985. In January 1985 the University Library at Wurzburn, Am Hubland, suffered water damage to its holdings. A small number of books were dried by the freeze-dry technique. It appears that the library authorities were disappointed with the results; it was expected that after freeze-drying the books would be ready to put in the stacks. In another case involving water damage to materials of the archives of Beta Film GmbH, Munich, some unbound journals and other files were frozen to prevent microbiological attack. No vacuum freeze-drying was intended to follow. Instead, the materials were partly dried after a period of time in cold storage, then easily separated for conventional pressing and blotting (46).

10.1.6 Holland

Reported in 1985. The Central Research Laboratory for Objects of Art and Science had recent experience in freeze-drying water-damaged books from an archives building which had its roof blown away in a storm. Laboratory equipment was which, after some difficulties, produced relatively good results. In another project, equipment of the type found in the food industry has been tested. The Central Research Laboratory expects to have similiar equipment of its own in the near future (47).

10.1.7 Norway

September 1985. Rare volumes stored in the cellar of the Kleist Library in Tronheim were damaged by water leakage. The books were stabilized by freezing. Meanwhile, the Archaeological Conservation Laboratory, Tronheim Museum, was asked for advice. Freeze-drying was recommended. Eventually, the frozen books were placed in a freeze-dry chamber of 40 kilograms (88 pounds) capacity. Each drying run lasted eight to 14 days depending on wetness; the project took 25 days to complete. Parchment covers were removed while wet and air-dried to prevent damage during freeze-drying. Some leathers were treated with a low concentration of polyethylene glycol 400 in water before freeze-drying for more flexibility after drying. At the end of the drying cycle, the paper of the books was slightly dehydrated. This was solved by placing the books in a room with 50 precent relative humidity. Some books were cockled but were smoothed out with light pressing. The freeze-dry chamber, housed at the Archaeological Laboratory, is used normally for treating artifacts of wood and leather (48).

10.1.8 USA

August 1975. On August 24th a cloudburst and subsequent floods inundated the library at Case-Western University in Cleveland, Ohio. Some 40,000 books, 50,000 maps and periodicals were soaked and damaged in the muddy water. Seven freezer trucks were loaded with 40,000 pounds (18,140 kilograms) of wet papers and sent to the vacuum chambers at McDonnell Douglas Aircraft Company at St Louis, Missouri, for drying (39).

January 1977. On January 20th, Langley Hall at the University of Pittsburgh, Pennsylvania, suffered an explosion and fire from seepage of natural gas into the basement. The science and psychology library suffered extensive damage. The books wet from the fire hoses froze in place. Some 3,000 were stabilized in a freezer to await restoration (39).

February 1978. The San Diego Aerospace Museum and Library in California was destroyed by fire, apparently set by juvenile arsonists, on February 22nd. Ninety percent of the library's books, periodicals, films, photographs, and art were lost. Four large boxes of wet library materials rescued from the flames were frozen in cold storage. Here, the frozen materials were held until dried in a vacuum chamber. The materials were returned in good condition (39).

Reported in 1980. The Fall issue of the American Archivist reports that the University of Baltimore used a space simulation chamber at Johns Hopkins Applied Physics Laboratory to save books and manuscripts damaged by water in the extinguishment of a fire. Several hundred books and documents were dried in the vacuum chamber (49).

Reported in 1980. The Winter issue of the American Archivist reports the use of space simulation chambers to dry out Kentucky State records damaged by flood waters of the Kentucky River in December 1978. Initially, microwave ovens were used to dry the damp records, but the process was abandoned as too slow. As a result, the General Electric Company, Valley Forge, Pennsylvania, and the McDonnell Douglas Aircraft Company, St Louis, Missouri, were contracted for the project to dry, fumigate, and sterilize the water-damaged records (50).

The same issue of the American Archivist reports that the Idaho Historical Society has begun work on an emergency preservation plan which will outline actions to take in case of fire or flood. An essential part of the plan is to make prior arrangements for transporting water-damaged materials to local freezer and storage space. Local contacts will be made for commitments of temporary freezer storage space, transportation, carrying containers, and other supplies such as plastic sheeting and dehumidifiers. The emergency plan anticipates the use of freeze-drying for the recovery of most materials (51).

Reported in 1981. The News Bureau of the Lockheed Missiles and Space Company, Sunnyvale, California, reported in March of this year the successful freeze-drying, in a space simulation chamber, of more than 4,000 valuable books from the Doe Library Annex of the University at Berkeley. The books had been damaged by a defective sprinkler system that caused water to flow down and over the book stacks. The books were quickly sent to cold storage for stabilization while arrangements were made for drying.

In the same issue of News Bureau is another report on the use of freeze-drying: The drying of $3 million in counterfeit currency found in a slough. The United States Secret Service needed the currency to use as evidence in court proceedings (52).

11. COMMERCIAL SOURCES FOR FREEZING AND DRYING

11.1 Cold Storage

The most obvious place to begin a search for freezing and cold storage facilities is the commercial section of the telephone book. Rentals or purchases of chest freezers or larger units are usually found in the Appliances section; space for freezing and storing under Cold Storage Warehouses. And,although other industries, small or large, which have their own cold storage facilities do not cater to outside requests, many will and have made exceptions in an emergency. Among such organizations are frozen food processors including retail and wholesale outlets, ice cream and frozen desserts companies and their wholesalers and distributors, dairy companies and their outlets, and ice makers. Even supermarkets may have space or idle freezers which may be available.

11.2 Freeze-Drying

In the case histories summarized above, some of the vacuum chambers operated by aerospace companies had either been idle or used sporadically. The reactivation of these facilities occured when help was sought for drying large numbers of documents or books which had been damaged by water. As a consequence, what began as a trial test of a new technique became so successful that it is now an established salvage service available on a commercial basis. The literature cites several entities in this category.

This "spin off" concept seems to be gaining ground among organizations that use vacuum chambers for other purposes: Taxidermy, freeze-drying of biological specimens, experimental and testing laboratories. Capacity, of course, is limited. Nevertheless, advertisements are beginning to appear in periodicals that offer reclamation of books and documents soaked by water leaks, firefighting, or floods. Cost estimates are available along with the service of a technician who will arrange the preparation of wet materials for treatment in a vacuum chamber (53).

11.3 Vacuum Chambers

There are several companies on the international market engaged in manufacturing vacuum chambers for freeze-drying. With experts ready to advise potential customers on the type of system best suited for specific needs, the companies can supply vacuum chambers for use in laboratories or in larger versions for industrial purposes. One example is the VirTis Company (54) of Gardiner, New York. Included in its line of products is a system normally used by taxidermists which is castor-mounted and completely self contained. The system can use chambers of various sizes, the largest being 35 inches (88.9 centimeters) in diameter and 68 inches (172.7 centimeters) deep. The basic unit, called "Preservator," will permit the lengthy dehydration of animals at room temperature. Although the system is recommended for taxidermists, it can be used for freeze-drying wetted archival and library materials.

Another organization, internationally known in the field, is Edwards High Vacuum (55) located in Crawley, England. EHV also manufactures its products in Marburg, West Germany; Milan, Italy; Grand Island, USA. Among its line is a small unit intended for freeze-drying of biological and pharmaceutical preparations which has been used for freeze-drying books. The unit is called the Modulyo 4K. It has a drying chamber made of perspex in cylindrical form which can be custom designed to hold up to 50 books.

Vacu-Systems Incorporated (56), Houston, Texas, carries in its line of vacuum chambers a mobile freeze-dry system called the MD 24. It has a chamber with capacity for about 100 books. The system sells for about $14,000 (1985 prices); rental for this unit runs about $1250 per month with a two month minimum rental.

12. COST OF VACUUM-DRYING AND FREEZE-DRYING

There is no universal method available for determining the cost of drying water-damaged archival and library materials in vacuum chambers. The estimates, generally based on the quantity of materials to be treated, may include a calculation of man-hours spent by technicians and operators of the chambers, amount of electricity used, supplies and materials, plus the profit factor. At the other end of the scale there are many cases of moderate to no costs, thanks to a desire, on the part of the owners of the chambers, to foster good will or to render a public service. And because the mission of most archives and libraries is of a non-profit nature many organizations often are willing to charge only the costs involved.

In the cases that follow, some prices quoted are old. A calculation upward will have to be made for inflation. The prices do not include transportation and storage. Nevertheless, they will convey a general idea of what vacuum chamber drying can cost as shown in specifically cited cases.

The recovery operations incident to the Charles Klein Law Library fire, in June 1972, were handled by a salvage company, Recovery Services International, which is a subsidiary of the insurance carrier, Insurance Company of North America. Since the library's collection was insured, the carrier wanted to recover as much as possible in order to lower the amount of the claim. To this end, the salvage company selected a vacuum chamber operated by the General Electric Space Simulation organization, Valley Forge, Pennsylvania, for freeze-drying the water-damaged books awaiting in a commercial food freezer storage plant. It was estimated that on the basis of the large amount of volumes involved, the cost would be under $2.00 for each book (32). At the same time, insurance officials at Temple University, site of the library, had calculated the cost of restoration would be about $4.00 per volume as opposed to $25.00 per volume for cost of replacement. However, it was recognized that many of the salvaged volumes could not be replaced at any cost (57).

The Stanford University Meyer Library flood, November 1978, involved over 50,000 volumes. The University had no commercial insurance, but was self insured for a specific sum of money. The use of the Lockheed Missiles and Space Company vacuum chamber for freeze-drying was estimated to be $40,000. However, the company decided to absorb the cost as a contribution to the University. The Modern Ice and Cold Storage Company where the volumes were stored in their frozen state absorbed some $8,000 worth of freezer space and labor. The Eastman Kodak Company donated $2,000 worth of film restoration. Various other businessmen contributed expertise, food, labor, trucks and supplies (58), all of which helped reduce the final overall costs of the recovery operations.

In January 1979, a burst water main flooded the Taylor Institution Library at Oxford. Some 200 volumes were taken in a frozen state to the Atomic Energy Research Establishment at Harwell where they were vacuum-dried in a laboratory chamber. It is interesting to note that this vacuum drying operation was the first successful attempt of its kind in England (59). There were no costs involved for the work done.

In January 1981, the fire and subsequent water-damage to the records of the Regional Office of Income Security Services in Winnipeg involved some 150 file drawers which, when emptied, filled over four hundred plastic crates used to freeze the documents. The drying process took place in a high altitude chamber used by the Canadian Forces Base at Winnipeg. The recovery costs estimated by the Income Security Services authorities were not to exceed $12,000. However, the Armed Forces contributed their efforts in the recovery of the documents as a public service. Hence, the final costs for the entire operation were considerably less (37).

In August 1985, an informal query on rates was made of a small company in the USA that specializes in freeze-drying of water-damaged documents and books. The following information was obtained: The cost of drying 100 frozen books, about textbook size, in a vacuum chamber with a capacity of 12 cubic feet (.34 cubic meters) is about five to six dollars a book (transport costs not included) (53).

At first glance, freeze-drying in sophisticated chambers of large quantities of wetted materials may appear high. However, if you weigh the costs of air-drying, hiring of additional personnel, requirements for space, rental or purchase of apparatus for control of humidity, cost of materials, loss of time, the risk of mold infection, against the costs of freeze-drying, the price begins to look reasonable.

A clue to where the cutoff point ends and the positive cost factor begins was provided recently in conference held by the American Institute for Conservation. One of the participants in a seminar, who has had considerable experience on the subject, felt that the cost factor per item factor becomes reasonable only when freeze-drying more than 2,000 books, or 3,000 to 4,000 manuscripts unless small chambers are used. However, these are less effective because they have less sophisticated equipment. Consequently, it takes longer to pull the vacuum, maintain and dry the contents (60).

13. LOW COST FREEZING AND DRYING IN AN EMERGENCY

13.1 General Considerations

The process that follows has as its key feature the use of a normal freezer for the stabilization of wet documents and books, followed by conventional air-drying. It is suitable in those situations where quantity of wetted materials is not great, and where help and time are available. The materials to be frozen and later air-dried should be selected by priority based on whether they are just damp or soaked, slightly or very damaged, and whether valuable and irreplaceable.

The freezing of a bundle or stack of documents should not be a problem. However, during the thawing and drying operation there may be a risk that soluble inks and colors will feather or run. As to books, the technique works well when limited to relatively modern editions. With older, larger, and heavier books the drying treatment becomes somewhat complicated; the weight of water can seriously damage their structure. For material printed on coated paper, the best and, perhaps, only method for salvage is to freeze while wet and freeze-dry.

13.2 Materials Required

If cleaning is required: plastic pails, sponges, a handy source of water, drainage; paper towels are always handy for this wet operation.

Preparation for freezing: work table, plastic sheeting for work surfaces (the plastic is useful for many things), freezer paper (Kraft or similiar if none available), tape, sturdy boxes or plastic milk crates if transporting is required.

Freezing: domestic chest freezer or other.

Air Drying: electric fans, hand-held driers.

Mold Prevention: thymol crystals (a fungicide) and a suitable colvent (ethanol, denatured alcohol) to make interleaving sheets; for this purpose unprinted newspaper stock or strong tissue.

13.3 Recovery of Water-Damaged Materials

13.3.1 Handling Wet Materials

The materials selected for freezing are taken to the area where cleaning, if required, will take place, or to the work tables for wrapping. Extreme care must be taken in handling the wet materials in order to reduce the risk of damage with its consequent high cost of repairs and restoration.

No attempt should be made to separate one or more documents from a soaked pile. If for some urgent reason a document has to be removed, cut a sheet of plastic a bit larger than the document to be removed. Place the plastic over the soaked document and with a fine tool (spatula, knife) lift one corner so that it adheres to the plastic sheet. Lift gently; the surface tension of the water will cause the document to stay with the plastic. To release, place both on an absorbent paper with face of document down; with tool hold corner of document down and lift the plastic sheet with care.

If an archival box is soaked replace it in situ with a fresh one if safe to do so. This will prevent the contents from spilling out.

Soaked books should not be opened or closed. To do so will cause severe strain on the structure of the book and damage to such elements as hinges and spines. A soaked book should be taken to the work area as is. Soaked covers should not be removed; they help support the text block and lessen the risk of damage.

13.3.2 Cleaning and Washing

Before freezing, it would be helpful to remove soilage from the water-damaged materials. Many times this is not possible because facilities are not available, or because of the urgency of the situation. In any event, if soiled books are kept firmly shut they can be dipped in running water (flowing slowly) and the soil gently sponged away. A pile of documents with soiled surfaces cannot undergo the same method of treatment. One way to remove soilage is to play a gentle stream

of water over the pile through a soft rubber or plastic tube than can be pinched for control of flow.

13.3.3. Wrapping and Packing

Wrapping supports wet materials; it keeps a bundle of documents or books from freezing one to the other. Wrap books singly; documents in stacks as thick as a book. If the freezer is nearby, place items there as each is ready. If at a distance, use cardboard boxes or milk crates for transport. Do not wait for large pile. The sooner stability takes place, the less risk of mold. (Note: Some conservators feel that with large quantities of soaked materials, complete wrapping is unnecessary; separation of items with freezer paper works well).

13.4. Freezing

These are freezing sources: Upright ice cream freezers, commercial frozen food lockers, domestic chest freezer, the freezing compartment of a domestic refrigerator. The literature recommends a freezing temperature of -20°F (-29°C). The first three units have this capability; the last, with higher temperatures, does not.

13.5. Air Drying

13.5.1. Picking a Work Area

The work area should be such that air-drying will be encouraged and mold infection discouraged. Much depends on the weather; summer or winter, humid and hot, cold and dry. Bower and Brandt (37) describe an operation where prevailing variables were used to produce a very satisfactory environment for air-drying a number of soaked files. These, still in their cabinets, were moved into the basement of a heated building. The winter weather outside ranged between -7° and -28°C (19.4°and -18°F). The heat in the building's basement was lowered as much as possible; the relative humidity reached 30 percent.

In summer, particularly if hot and humid, the task may be more difficult. If the work area has air conditioning that can maintain a temperature of about 18°C (64.4°F) and a relative humidity of 50 to 60 percent, mold growth can be held in check. If air conditioning is not available, open windows and doors to encourage circulationand get rid of stagnant air; use electric fans to assist. If the humidity remains at high level, the use of dehumidifiers may be in order. These are available at appliance outlets and, in some cases, can be leased. When dehumidifiers are used, close doors and windows; use electric fans for circulation. (Note: An inexpensive hand-held instrument, the sling psychrometer, will give sufficiently accurate

readings of temperature and relative humidity to eliminate guesswork).

The working area will need several tables for the operation; working surfaces should have protective plastic sheeting. Have sufficient electrical outlets for fans, air driers, dehumidifiers, and other equipment. Use heavy duty, well-insulated cable extension cords.

13.5.2 Drying Documents

Frozen documents can be dried with relative ease. If blotting paper (newsprint stock or paper towels will do) is available place on the work table. Unwrap each bundle and place on the absorbent. Electric fans blowing over the documents will circulate the air and help thawing and evaporation. Hand-held driers will hasten drying. As the topmost document drys sufficiently to be handled safely, lift off and place on another surface for further drying. By removing the top document, the next one is better exposed to the air for evaporation. When it becomes safe to divide the pile into smaller ones, drying will also be speeded up.

With a bit of ingenuity, any number of methods can be found as drying surfaces for documents as they are lifted from a pile: tiers of plastic screens secured to wooden uprights, nylon cords over which the documents are draped, the lawn outdoors if conditions permit.

Soaked absorbent paper and other wet scrap materials should be removed from the work area in order to keep the humidity level down.

13.5.3 Drying Books

Book thawing followed by the evaporation of its moisture is a slow process. It takes time to dry from the outside in. Place the book on its side on blotting paper or other suitable absorbent. Use electric fans to assist thawing. Do not use heated driers since the drying rates of the components that make up the book vary; unecessary strain and tension should be avoided. When the book is thawed stand upright (on its head to compensate for shelf sag), open covers slightly for support; do not fan out. As the book dries it can be opened a bit more for additional area exposure to air. Change absorbent often; remove soaked papers from room.

13.6 Pressing

Normally, a book contains about seven percent moisture. It is never totally dry even when reposing on a shelf for a long period. Documents comport themselves in the same way. However, neither would respond to pressing with so little moisture. On the other hand, when thawed books and documents are air-dried to the point where they feel "dry" to the touch, they usually have more than the amount of mosture required for response to pressing.

Documents with wrinkles, cockling, or creases can be flattened by pressing between boards with a light weight on top. Press several at a time; separate each with silicone paper (wax paper will do). As to books, place each on its side on a flat surface; with thumbs on the front edge press inward gently to form a rounded back. Place book between pressing boards with light weight on top. Do not stack books for pressing.

13.7 Prevention of Mold Infection

Should it appear that conditions are favorable for mold infection of books, a practical method of applying an inhibitor is by interleaving with fungicidal sheets. The method is particularly useful when the amount of materials to treat is not great.

In this technique sheets of paper (newsprint stock, strong tissue) are cut a bit larger than the item to be treated. The sheets are dipped in a thymol solution. For this purpose, a 10 to 15 percent solution of thymol crystals in ethanol, acetone, industrial denatured alcohol, or trichlorethane (one pound of thymol crystals in one gallon of solvent, or 450 grams of the crystals in about four liters of solvent) is recommended (5). The sheets are placed on a surface protected with plastic sheeting for drying (which is rapid). To store, put sheets in a sealed container or wrap in plastic since thymol is very volatile.

Interleave books in their upright position when dry enough to open slightly without the risk of damage. Place the thymol sheets in the text block at intervals of about 25 millimeters (one inch). Change frequently. Do not interleave too much lest the spine of the book suffer damage. Because of their construction, books are easier to interleave. However, there is no reason why the same technique cannot be used on documents.

Care must be exercised in preparing the thymol solution because of the toxicity of the products. Rubber gloves should be used along with a painter's respirator, and goggles. If the fungicidal sheets can be prepared outdoors, so much the better.

14. WHAT FREEZE-DRYING WILL NOT DO

Freeze-drying is a salvage process; salvage in the sense that its purpose is to save water-damaged archival and library materials from destruction. This is done by freezing to stabilize, then sublimate to dry. It will not return to pristine condition any books or documents which were in a damaged state at the moment they were frozen for subsequent drying. Freeze-drying will not press out cockled or wrinkled sheets and pages, nor straighten out warped covers. These will be locked in that position on drying. It will not release the pages of a book printed on coated stock if blocking has set in before freezing.

It will not remove discoloration, tide marks, or stains that were there at the start. Freeze-drying will not press out vellum or leather. And as pointed out, it will not destroy mold spores.

In short, apart from the end purpose of the process, frozen materials come out of the vacuum chamber in just about the same condition as they entered. At the end of the drying cycle some will look surprisingly good; others will be scheduled for minor repairs. Some may have to bear the high cost of restoration; some may have to be discarded or replaced. In any case, the fault is not in the freeze-dry process. It is still the most effective method known for the physical, chemical and biological stabilization of water-damaged archival and library materials, especially when large quantities are involved and time is of the essence.

15 COOPERATIVE APPROACH TO THE USE OF FREEZE-DRY CHAMBERS

15.1 General Considerations

The price of a vacuum chamber with the capacity to freeze-dry approximately 100 books or the equivalent volume of documents is relatively modest: cheaper than the price of some automobiles. However, it would make little sense for individual archives or libraries to purchase such a machine, install it, train an operator, then wait for a disaster to occur. There is no economic logic to the plan. With this thought in mind, some alternatives are suggested which may be helpful in finding a more viable approach with more practical and economic sense.

15.2 Regional Cooperation

It may be of interest to mention a highly successful example of regional cooperation in sharing the financial costs of services and facilities required to restore, preserve, and maintain books and documents under the custody of several institutions. In 1972 six New England states drafted and approved an interstate library compact that created the New England Document Conservation Center (61) in North Andover, Massachusetts.

The compact provided an agreement that each state government would contribute funds to the NEDCC, allow membership fees, and accept grants from foundations as a non-profit organization. Members such as public libraries, state and local archives, private but non-profit educational institutions, would be entitled to use the services and facilities of the NEDCC on an "at cost" basis. Since 1972 several changes have been made. Membership has expanded, the name changed from "New England" to "North East" and its services open to the public sector.

As a regional organization the NEDCC "owns" several pieces of high cost equipment, some mobile, that many libraries or archives could afford or want. This equipment is available to a vast clientele when needed.

15.3 Cooperation With Public Institutions

In most countries the answer to the accessibility of vacuum-drying and freeze-drying facilities might be found in a cooperative understanding between the smaller and more numerous state funded archives and libraries, and the larger, better endowed public institutions. In the latter category are national archives and libraries, state universities, museums, scientific organizations, and certain of the armed forces. Some of these entities either have the potential for operating a vacuum chamber, or actually have and operate one.

In the case of entities with a potential, two examples can be cited. The first is the National Library of Austria in Vienna. It is anticipated that a freeze-dry chamber will be installed in the library's restoration laboratory under the supervision of a conservator-scientist. The chamber will be used not only for drying water-damaged books and documents, but for other conservation techniques as well. The situation at the National Library is ideal: A prestigious institution with a technical staff totally familiar with paper, leather, parchment; a place where the comportment of sizes, adhesives, inks and dyes, in the presence of water is known. The whole of this knowledge can be useful in the scientific investigation and application of vacuum-drying and freeze-drying techniques to water-damaged archival and library materials.

Another organization with similiar potential is the Central Research Laboratory for Objects of Art and Sciences in Amsterdam. Its scientists have had experience in the freeze-drying of water-damaged books using laboratory equipment. The institution has also worked with machines of the type used in food industry. It is their hope for the future to have a vacuum freeze-dry chamber for experimental purposes. Undoubtedly, the chamber will also be available for use in any emergency.

Such public institutions appear to be the most logical custodians of freeze-dry chambers. Both are oriented toward the preservation and conservation of cultural objects; both are rich in scientific expertise in this area. Furthermore, the appropriation of funds can be justified by the dual mission of the chamber: Laboratory experiments and testing, its availibility for the salvage of water-damaged archival and library materials.

As to the second case, there are many state universities and scientific centers that possess freeze-dry chambers of various sizes. These could serve to dry relatively small amounts of wetted materials. However, even with agreement to help in an emergency, it is understandable if there is reluctance on the part of a department to tie up its personnel and chambers to dry a bunch of frozen documents or books that had the misfortune of getting wet. Nevertheless, if the materials are in cold storage there is time to wait.

Another public entity that normally has vacuum chambers is a nation's armed forces, specifically, the air force. This arm requires such chambers for the study of space medicine and related experiments, particularly for the observation and testing of the physiological behavior of the human body at high altitudes. The supervision of these tests is usually under medical personnel and specialized technicians. As cited in this study, vacuum chambers have already been used in drying large amounts of wetted archival materials. Since these facilities are in the public domain, there is a possibility that interdepartmental consent could be granted for their use in an emergency. And if a nation has a public funded space program, this is another possibility to explore.

15.4 Commercial Cooperation

The fact that the majority of archives and libraries are non-profit organizations with a selfless mission has, in many cases, prompted owners of commercial vacuum chambers used in a disaster to absorb the costs involved. While it might be asking to much for an extension of this courtesy ahead of time, there is no reason why an attempt cannot be made to offer payment for emergency services on a cost basis. Commercial companies that process food, and aircraft companies engaged in space activities should be kept in mind for drying sizable quantities of water-damaged materials since their chambers are of large capacity.

16. AN OPEN FORUM ON FREEZE-DRYING

16.1 Questions and Answers

In this imaginary open forum, thequestions posed (to which answers are provided) were taken from a number of actual conferences on conservation of books and documents, seminars on disaster preparedness, and panels discussions on freeze-drying. The "Questions and Answere" have been placed in this study to expand on a number of points related to freeze-drying which may have been touched upon or briefly mentioned in the text.

16.1.1 In simple terms, what is the difference between freeze-drying and plain vacuum-drying?

Actually, the difference is in the amount of pressure developed by the pumping system. With the capacity required to reach levels of relatively high vacuum, freeze-drying becomes possible. The frozen materials in the chamber are sublimated; they pass from water as a solid to water as a vapor without going through the liquid stage. In vacuum-drying, the pumping system produces lower values of vacuum. Here, the materials are placed in the chamber in their wet condition and are evaporated, that is, water as a liquid passes to water as a vapor. In freeze-drying, the water vapor is normally trapped by a refrigerated condenser; in vacuum-drying the water vapor can be trapped by this method or others such as steam ejection.

16.1.2 Is freeze-drying expensive?

Yes and no. Freeze-drying becomes expensive when the vacuum chamber used for drying is large and sophisticated, and the amount of water-damaged materials is small. If the quantity is relatively large, freeze-drying becomes inexpensive when you calculate in terms of time, personnel, space, materials and supplies required to the dry the materials by traditional methods. Freeze-drying in a smaller vacuum chamber is, of course, less costly although capacity is limited and drying takes longer. In either case, if it appears that your materials will require expensive restoration, but can be replaced by some other format, there is little to gain by freeze-drying.

16.1.3 What about the cost of vacuum-drying?

Vacuum-drying is somewhat less expensive than freeze-drying; one reason is that you do not freeze wetted materials beforehand; the chamber is less sophisticated. However, what is gained in economy is lost in what freeze-drying supplies: stabilization, time, the the advantages of sublimation over evaporation. The conditions of costs that apply to freeze-drying - large quantities of materials in large chambers are less expensive - also apply to vacuum drying.

16.1.4 Can wet (non-frozen) materials be freeze-dried, and conversely, can frozen materials be vacuum-dried?

Yes, to both parts of the question. However, when wet (non-frozen) materials are placed in a freeze-dry chamber, as the initial water "rushes" off, they will freeze. When this happens there is a possibility that bound books may come apart and heavy calendered paper puff or split. It is best to freeze the wet materials first. In regard to frozen materials placed in a vacuum-dry chamber, during the drying cycle the ice will melt, become liquid, and carry the risk of possible migration of colors, stains, and inks (62).

16.1.5 Which is the better system, freeze-drying or vacuum-drying?

Remember that in freeze-drying the materials are frozen first, then sublimated in a freeze-dry chamber. In vacuum-drying the materials are placed in the chamber in a wet condition for drying by evaporation. In general terms, the tendency seems to favor vacuum-drying for office records when there is no imminent danger of mold attack and a chamber is available immediately. On the other hand, for books and their special structure, freezing followed by sublimation is the preferred method. Books printed on coated stock cannot be salvaged by the evaporation process; sublimation in a freeze-dry chamber is the only way.

16.1.6 Shouldn't time be taken to sort or weed out wet materials prior to wrapping and crating for freezing?

A moot question. Much depends on the situation and a variety of factors: Amount and importance of the items, degree of damage, climatic conditions, help available, time, and so on. A case in point, where the research value of water-damaged materials outweighed a decision to weed out in order to save time and money, is the action taken at the Meyer Library flood, Stanford University (4). Involved were some 50,000 volumes of a general research collection in humanities and social science. The materials had been collected for ten years as part of a collection development program that was a prime resource for faculty and students; their value weighed heavily in the decision not to sort or weed out.

In another case, water-damaged items in a library basement after a fire were exposed to conditions favorable for rapid mold attack. So, some 30,000 volumes and other vulnerable materials were frozen and placed in cold storage without sorting or weeding out (31). Later, it was found that some items could have been replaced at a fraction of the cost. However, this was not considered during recovery operations because of the prevailing conditions.

16.1.7 Shouldn't wet materials be cleaned of soilage prior to freezing?

It would be desirable, but not if it requires a loss of precious time and risk of mold infection to organize crews and setup cleaning arrangements. However, there will be occasions when there is no choice but to go ahead with some cleaning. Each case will have to be judged by its own merits.

16.1.8 What is the best way to wrap wet materials for freezing?

The primary reason for wrapping wet materials is to keep them from freezing into large, unwieldy blocks of ice. Some people use freezer paper on books or bundles as if it were arm sling; no need to fold over. In other cases, if the wet materials are placed in boxes or crates, a few at a time, and will not be removed for either the freezing or freeze-dry operation, there is no need to wrap or separate with freezer paper.

16.1.9 At what temperature should water-damaged materials be frozen and stored?

Freezing and storage temperatures which have been used vary from about -20°C (-4°F) in a chest freezer for a few soaked books to -30°C (-22°F) in cold storage for larger quantities of wetted materials. Waters (5) recommends a freezing temperature of -20°F (-29°C); lower temperatures will do no harm. In Stanford University's recovery operation, that temperature was used in a cold storage facility (63).

16.1.10 Wet books and documents undoubtedly swell and expand with freezing. Doesn't that cause damage?

Saturated materials do swell even more on freezing but this additional thickness does not add to the damage already caused by water. In studies conducted by the Research and Testing Office of the Library of Congress, there was no evidence found that freezing causes damage of cellulosic and proteinaceous materials (5).

16.1.11 Do you need a special chamber for freeze-drying books and documents?

No. Generally, the design, size and original purpose of the chamber makes no difference. Huge vacuum chambers made to test gear and equipment used in space programs have successfully freeze-dried large quantities of water-damaged books and documents. Small chambers, such as those used by taxidermists, can freeze-dry the same materials; the quantity is smaller because the capacity of the chamber is much less.

16.1.12 How do you know when the materials in a chamber are dry?

Methods for determining the level of dryness vary. When heating strips are used, temperature probes are inserted in several of the frozen materials and the temperature is plotted. As the dry state of the materials is reached, the temperature curve moves to the temperature of the heater strips (63). Another method is to plot weight loss. The frozen item is weighed before it is placed in the vacuum chamber; at several intervals of time during the drying cycle the test sample is weighed. A state of dryness is reached when the weight loss reaches a plateau (36).

The Aquaboy, a book probe manufactured by K.P.Mundiger, Wartenburg, West Germany, is a useful instrument for measuring water content of paper. This probe has two metal prongs, resembling a tuning fork, that are placed flat on the exposed page of a book; the book is then closed. A dial on an instrument to which the prongs are attached registers the absolute humidity inside the book (35). A reading of five to seven percent indicates normal moisture content. Note that this percentage is not "relative humidity" as some people think. In technical jargon, the moisture content of paper is called "absolute humidity" which is the actual weight of water vapor in a unit of moist air in grams per cubic centimeter.

And a final method, though not very accurate, is to open the chamber from time to time and touch the materials to test for dryness.

16.1.13 Is there a risk of overdrying and, as a consequence, run the risk of damage to the materials?

Yes, to both parts of the question. Books that come out of a vacuum chamber in this condition, which is usually the case, must not be opened and closed because the pages and structure is so fragile. Documents must also be handled with care. The solution to this problem is to get some moisture back into the paper. This can be done by placing the dry materials in a room with normal conditions of relative humidity; about 55 to 60 percent. It can take up to three or four weeks for the paper to equilibrate with the atmosphere.

One research center solves the overdrying problem with a series of humidity chambers of varying humidities and temperatures for the introduction of moisture into the books. Pressure is applied to reach the approximate water content they had originally (13).

Paper is like a sponge. Water content under normal conditions is between five and seven percent by weight. However, this level varies proportionally with the amount of relative humidity in the air. For example, at 80 percent relative humidity, paper absorbs between 9 and 14 percent water (leather absorbs 18 to 20 percent); at 65 percent the various parts of a book can vary 6 to 9.5 percent of water content (6).

16.1.14 Can parchment and leather be freeze-dried?

Both can be stabilized by freezing then vacuum freeze-dried. However, at some point, particularly with rare or valuable items, the advice of an experienced conservator should be sought. This is especially important where parchment is concerned since this material is altered structurally when wet. The best moment for seeking advice may be after stabilization of the materials, but not after the freeze-dry cycle. Only an expert should carry out the recovery work.

In relation to the question, French conservation scientists conducted some freeze-drying tests under laboratory conditions on paper, leather, and parchment. These were immersed in water, frozen, then freeze-dried in a laboratory vacuum chamber. One of the observations made was that freezing can be used as a method of storage for the three materials. The results of freeze-drying each material follows: The technique is wholly indicated for drying paper. Leather can be freeze-dried without great risk. On the other hand, parchment poses distinct problems since water causes internal changes that result in thickening and a decrease in elasticity. The scientists point out that these changes are reversible. After freeze-drying, the parchment samples were placed in the hands of expert restorers at the Bibliotheque Nationale who used moisture and pressing to recuperate suppleness, color and thickness (64).

16.1.15 In the freeze-drying process, heat is sometimes applied to the frozen materials. First of all, why is it done and doesn't the heat harm the materials?

Heat is applied to the frozen materials in order to hasten the sublimation of the ice; it is normally done when large quantities of materials must be dried. The temperatures applied by flexible rubber heaters on shelves in the chamber are not excessively high. In one case (32), the temperature starts out at 32°F (0°C) on books and will not get warmer than 80° to 85°F (26.5° to 29.4°C). In another case (14), with frozen materials heated by aluminum foil heaters, the book temperature was never higher than 100°F (37.8°C). One research center uses 40°C (104°F) as the maximum shelf temperature for documents.

16.1.16 Once the materials come out of the freeze-drying chamber can they go straight back to their shelves?

The reply must be qualified. In the first place, the materials that come out of the chamber might be overdried and require rehydration to a normal level before they can be handled safely. Secondly, some materials may have gone into the chamber in a damaged condition; they will come out the same way and require repairs or restoration. Thirdly, the stacks where the materials were water-damaged may not be in any condition to receive them and may need rehabilitation.

An ideal situation would be to take the materials to a place where they can return to normal humidity content, where they can be pressed, repaired, or restored, and be under constant vigilance for mold infection (some books and documents come out of the chamber partially dry).

16.1.17 Would it not be less costly to replace water-damaged materials than freeze-dry?

Generally, yes. If books, for example, have been soaked in water to the point where they have swelled, split their component parts, warped their boards, it would be much cheaper to replace, if such can be done, than to restore.

16.1.18 It is generally known that freeze-drying will not destroy mold spores, but what about insects?

Actually, freezing is deadly for some destructive insects. An infestation of beetles, the Anobiidae, at Yale University's Beincke Library, was controlled by freezing the affected books. The university biologist who recommended this method had been using it to kill insects, their eggs, and larvae for exhibits and laboratory purposes. As far as known, the method had not been used for infested library books (65).

16.1.19 How do you go about freezing insects?

Simply put the infested material in a freezer. At Yale, tests were first run in domestic freezers. When they proved

successful, a 368 cubic foot (10.3 cubic meters) blast freezer (a type of rapid freezing model) was obtained in which the infested books were frozen at -30°C (-22°F). First, the books were sealed in polyethylene plastic and put in the freezer for 72 hours. They were taken out and permitted to dry overnight. The only moisture noted was on the outside of the plastic envelope (65).

16.1.20 Why weren't the infested books fumigated?

They could have been. Nearly 40,000 volumes were involved. Yale personnel opted for the cleaner, safer, although slower method of freezing (the freezer had capacity for about 400 books at a time). Fumigation would have required that the entire building be vacated, sealed, and be left that way for several days. Spraying was ruled out because of possible harm to the books; vacuum fumigation was also considered, but also ruled out because of high costs (65).

16.1.21 What should you do if mold is widespread before the wet materials can be frozen?

Fumigation is the first thing that comes to mind. However, there is no clear-cut answer at the moment; opinions vary. The controversy centers on health hazards, and the long-term effects of the toxic products used (ethylene oxide, among others) to destroy effectively fungal growth and spores (also insects and eggs). And for deep, penetrating destruction a vacuum chamber, fixed or portable, must be used. Many countries do not have these sophisticated machines, nor the professionals required for the use of fumigants that are highly toxic to people.

Nevertheless, there are lower scaled, albeit less effective, steps recommended in the literature: fogging with thymol crystals dissolved in a suitable solvent for use in specific cases. For example, when the damage is extensive in order to destroy spores already present, and prevent growth of new ones (3). Or, in cases of large collections where access to them has not been possible for several days, the same fogging technique is recommended (5). Because of health hazards, fogging should be done by a professional fumigator.

But what action can you take if fumigation and fogging facilities are not immediately available? There seems to be agreement among a number of conservators (based on informal talks) that in such a case, and faced with widespread growth on wet materials, they would do the following: First, not try to brush or rub off the growth; it will leave a difficult-to-remove smear. Next, prepare the materials for freezing to stabilize them, then, at the appropriate moment, freeze-dry. At the end of the drying cycle, the mold can be brushed off (use masks for people; brush outdoors) with no problem.

To take the problem a step further, the same conservators were asked to recommend a course of action if neither fumigation or freezing facilities (vacuum chambers included) were available. They were unanimous in suggesting that the wet materials be removed from their damp venue and placed in a cool, dry place for air-drying in the traditional manner. They would use any and all electrical materials possible to speed up drying and maintain strict control of the environment. Their final recommendation is, at some point, if at all possible, to fog or fumigate.

16.1.22 Can mold-infected materials be fumigated in the same chamber where freeze-drying takes place?

Yes. A professional who knows the safety and health requirements should do the job since the fumigant used is toxic, and most places have strict air pollution standards when the moment for venting takes place.

Waters (5) suggests, as an additional preventive measure, that after sterilization the chamber be fogged with a 12 percent solution of thymol crystals in trichloroethylene. This treatment is a fungicidal buffer of temporary duration, but offers a high degree of resistance to further attack by mold.

16.1.23 If freeze-drying seems to destroy the visible growth caused by mold spores, why is it so necessary to sterilize and fog with a buffer? Why not put the dry materials in proper storage?

For what it is worth the following case is cited. After the Military Recrods Center fire in St Louis, Missouri, further recovery work was done with materials that had remained damp for four months. Mold had inevitably set in. However, instead of fumigating the infected materials, they were placed in the vacuum chamber facilities of the McDonnell-Douglas Space System Laboratory. The drying treatment arrested the development of the mold; the materials were then placed in an air-conditioned environment rather than bear the cost of sterilization (39).

16.1.24 Some institutions have photographic materials in their holdings. Can they be frozen and freeze-dried?

Generally speaking, yes. However, the recommended procedure for recovery of water-damaged photographic material is to leave their treatment to a professional laboratory (some, for example, Eastman Kodak, have emergency services for that purpose). There are several sources that describe the methods used to stabilize the materials temporarily for transport to the nearest laboratory (66,67,68).

Briefly, the materials are placed in lidded plastic pails which are filled with clean, cold water. The materials (black and white photographs and film) last in water about 72 hours

before the emulsion separates from its support. Color materials are handled the same way except that the color layers will separate in some 48 hours.

Leaving the recommended procedure aside for the moment, there are cases on record where photographic materials were frozen then freeze-dried with satisfactory results. For example, after the Corning Museum Library flood, several thousand books were prepared for freezing and subsequent freeze-drying. During this operation many films, negatives, photographs, and slides were inadvertently packed along with the books. After the drying process, the photographic materials were discovered and found to be in better shape than the materials that volunteers tried to recover locally (30).

Another case. During the recovery operations incident to the Stanford Meyer Library flood, samples of microcards along with silver halide photographs, diazo and vesicular microfilm, and some computor tapes, were immersed in water for two hours then frozen at -15°f (-26°C) for 48 hours. The samples were then freeze-dried. The microcard disintegrated but the film itself was undamaged; the computor tape recovered well; the microfilm had minor spotting but no loss in text; the photographs were slightly cockled but easily pressed; cinema film in a roll in its cardboard container recovered well (4).

Hendricks and Lesser (66) did some tests on still photographic negatives and prints in black and white (and color), which along with other types were immersed in water then dried by various methods. Their preferred method of drying, if personnel and time are available, is first, drying by air, then freeze-thaw-dry by air, lastly freeze-drying in a vacuum chamber. They stated that wet collodion glass plate negatives must never be freeze-dried; none will survive.

17. A FINAL WORD: DISASTER PREPAREDNESS PLANNING

17.1 Why Prepare for a Disaster?

In the aftermath of some disasters, an earthquake, for example, where a building's archival or library materials are strewn about, time is of no importance; there is plenty of it to clean the mess, and make the necessary assessments and evaluations. But when a calamity is accompanied by water-damage, a different set of rules applies. You have only hours at your disposal to take appropriate action to retard physical and chemical deterioration to cellulosic materials, and prevent biological infection from setting in. This is where preparedness planning comes into the picture. A well-prepared plan that permits the orderly deployment of personnel to their preassigned tasks and responsibilities for the execution of recovery operations, can convert a potentially chaotic situation to one you can classify as a lower level emergency.

17.2 What a Disaster Preparedness Plan Contains

A Disaster Preparedness Plan is no more than a set of instructions to tell people what to do upon the discovery of a disaster. It is drafted, discussed, coordinated, and finalized before and not during a disaster; it is kept up to date. It lists names and agencies to call in an emergency, action to take while awaiting help, appropriate methods for recovery of water-damaged materials, sources of supplies and materials, location of freezing and cold storage facilities (freeze stabilization could become the most important element in the plan), vacuum-drying and freeze-drying facilities, names and where to contact conservators, chemists, electricians, plumbers, fumigators, etc. In short, all those whose help might be required.

Recommended reading on disaster planning and preparedness can be found under REFERENCES. See 5, 8, 69, 73, 74, 75.

17.3 Prevention: Central to Disaster Preparedness

Disaster preparedness is incomplete if preventive measures are not included. Such measures contain the systematic inspection of those elements in a building that are potential risks or hazards to the integrity of an archival or library collection. For this purpose, a method can be developed - inspection sheets, for example - to flag the elements in the two specific areas that usually constitute a risk: Fire Hazards and Water Hazards. Another area that must bear close and periodic examination is the condition and adequacy of firefighting gear and equipment.

Under Fire Hazards check such items as security of inflammable materials and trash, condition of electrical systems, appliances, heating system, fuel deposits, etc. Under Water Hazards check potential dangers such as drainage and supply lines, steam pipes, water tanks, basins, sinks; condition of

roof, gutters, downspouts, exterior water mains, drainage, etc. An inspection sheet can be devised to inspect and record the condition of the fire prevention system and include inspection of fire extinguishers, detectors, alarms, etc.

Recommended reading on preventive measures can be found under REFERENCES. See 39, 70, 71, 72, 74, 75.

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